

FORM PTO-1390
(REV. 12-2001)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

MR2833-14

U.S. APPLICATION NO. (If known, see 37 CFR 1.5

10/069996

INTERNATIONAL APPLICATION NO.

PCT/US00/08943

INTERNATIONAL FILING DATE

5 April 2000

PRIORITY DATE CLAIMED

10 September 1999

TITLE OF INVENTION **QUANTITATIVE IMAGING OF DIELECTRIC PERMITTIVITY
AND TUNABILITY**

APPLICANT(S) FOR DO/EO/US

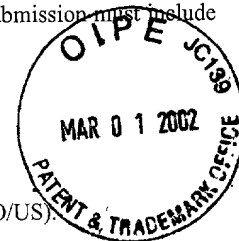
Steven Mark Anlage, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☐ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☒ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☐ Other items or information:



U.S. APPLICATION NO (if known, see 37 CFR 1.5) 10/069996		INTERNATIONAL APPLICATION NO PCT/US00/08943		ATTORNEY'S DOCKET NUMBER MR2833-14	
--	--	--	--	---------------------------------------	--

21. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1040.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$890.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS PTO USE ONLY <div style="display: flex; justify-content: space-between;"> \$ 890.00 </div>	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	29 - 20 =	9	x \$18.00	\$ 162.00	
Independent claims	6 - 3 =	3	x \$84.00	\$ 252.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ \$280.00	
TOTAL OF ABOVE CALCULATIONS =				\$1,304.00	
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				+ \$ 652.00	
SUBTOTAL =				\$ 652.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$ 652.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +				\$ 40.00	
TOTAL FEES ENCLOSED =				\$ 692.00	
				Amount to be refunded:	\$
				charged:	\$

a. ☒ A check in the amount of \$ 692.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

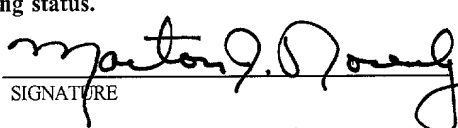
c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 18-2011. A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Rosenberg Klein & Lee
3458 Ellicott Center Drive, Suite 101
Ellicott City Maryland 21043


 SIGNATURE
 Morton J. Rosenberg
 NAME
 26, 049
 REGISTRATIONNUMBER

10/069996

PCT/US00/08943

JC13 Rec'd PCT/PTC 01 MAR 2002

Statement Concerning Non-Prejudicial Disclosure or Exception to Lack of Novelty

Due to possible disclosure not earlier than April 5, 1999, the applicant respectfully requests that the subject international application be granted the respective provisions under national laws concerning Exceptions to Lack of Novelty in each of the designated countries. This is not an admission that the subject invention is not novel. Exception to Lack of Novelty is hereby requested for purposes of disclosure and precautionary measures.

10/069996
JC13 Rec'd PCT/PTC 01 MAR 2002

Quantitative Imaging of
Dielectric Permittivity and Tunability

ART 34 AMDT

***Statement as to Rights to Inventions Made Under
Federally-Sponsored Research and Development***

5 This invention was made with government support under Contract
Numbers ECS 9632811, DMR 9632521 and DMR 9624021 awarded by the
National Science Foundation. The U.S. Government has certain rights in this
invention.

Background of the Invention

10 ***Field of the Invention***

The present invention relates generally to imaging, and more particularly
to measuring dielectric properties using a near-field scanning microwave
microscope.

Related Art

15 Dielectric thin film research has become increasingly important as the
demand grows for smaller, faster, and more reliable electronics. For example,
high permittivity thin films are under study in order to fabricate smaller capacitors
while minimizing leakage. Low permittivity materials are being sought to allow
smaller scale circuits while minimizing undesirable stray capacitance between
20 wires. Nonlinear dielectrics, which have a dielectric permittivity which is a
function of electric field, are being used in tunable devices, particularly at
microwave frequencies. Finally, ferroelectric thin films are a solution for large-
scale, non-volatile memories.

AMENDED SHEET

ART 34 AMDT

-2-

5 All of these dielectric thin film technologies demand high-quality,
homogeneous films. However, this goal is often difficult to achieve because of
the complicated fabrication process involved. Consequently, it is important to
have a set of reliable techniques for evaluating thin film properties, such as
permittivity and tunability. A number of different techniques are available. One
10 standard low-frequency (≤ 1 MHz) method uses thin film capacitors to measure
the normal and in-plane components of the permittivity tensor. Another uses
reflection measurements from a Corbino probe. Still another method provides
microwave measurements of permittivity by using transmission measurements
through a microstrip structure. However, these techniques average over large
15 areas and involve depositing thin film electrodes which itself can alter the
properties of the sample. Dielectric resonators have been used as well, but also
have the problem of low spatial resolution. More recently, near-field microscopy
techniques have allowed quantitative measurements with spatial resolutions much
less than the wavelength. These techniques use a resonator which is coupled to
a localized region of the sample through a small probe and have the advantage of
being non-destructive. However, it is still difficult to arrive at quantitative results
and maintain high spatial resolution.

20 Therefore, what is needed is a non-destructive, non-invasive, system and
method for imaging quantitative permittivity and tunability at high spatial
resolution.

Summary of the Invention

25 The present invention meets the above-mentioned needs by providing a
system, apparatus, and method for quantitatively imaging the dielectric properties
of bulk and thin film dielectric samples. Permittivity and dielectric tunability are
two examples of dielectric properties capable of measurement by the present
invention.

AMENDED SHEET

12-11-2001

5 The system uses a near-field scanning microwave microscope (NSMM).
10 The NSMM is comprised of a coaxial transmission line resonator having one end
coupled to a microwave signal source and the other end terminating with an open-
ended coaxial probe. The probe, which has a sharp-tipped center conductor
extending beyond the outer conductor, is held fixed while the sample is raster
scanned beneath the probe tip. A spring-loaded cantilever sample holder gently
presses the sample against the probe tip with a force of about 50 μN
(microNewtons). A feedback circuit keeps the microwave signal source locked
onto a selected resonant frequency of the microscope resonator. Because the
electric fields generated by the microwave signal are concentrated at the probe tip,
the resonant frequency and quality factor of the resonator are a function of the
sample properties near the probe tip. Once the microwave signal has been
applied to the sample through the probe tip, it is reflected back through the
system. The feedback circuit is then able to receive the reflected microwave
signal from the coaxial transmission line resonator and calculate a resonant
frequency shift. The resonant frequency shift value is then stored in a computer.
The computer also controls the scanning of the sample beneath the probe. To
obtain quantitative results, the system uses calibration curves to exhibit the
relationship between the calculated resonant frequency shift data values and the
dielectric properties of a sample.

20 The invention described herein has the advantage of being able to provide
quantitative results for samples on a length scale of about $1\mu\text{m}$ or less. This
allows for the measuring of sample sizes relative to the actual environment in
which they will be used.

25 The invention also has the advantage of providing more accurate
quantitative results because the sharp protruding center conductor is represented
as a cone during modeling.

AMENDED SHEET

12-11-2001

Brief Description of the Figures

5 The features and advantages of the present invention will become more apparent from the detailed description set forth below and the following figures in which like reference numbers indicate identical or functionally similar elements. Additionally, the left-most digit of a reference number identifies the drawing in which the reference number first appears.

FIG. 1 illustrates the general structure and functionality of an embodiment of the present invention.

10 FIG. 2 illustrates the use of a spring-loaded cantilever sample support according to an embodiment of the present invention.

FIG. 3 is a diagram of a quantitative modeler used in an embodiment of the present invention.

FIG. 4 illustrates a resonant frequency shift detected by measurements taken when a sample was present versus when a sample was not present.

15 FIG. 5 shows a perturbation formula used in an embodiment of the present invention.

FIG. 6 is a diagram of the electric equipotential lines for a bulk sample according to a quantitative model of the present invention.

20 FIG. 7 is a diagram of the electric equipotential fields for a thin film sample according to a quantitative model of the present invention.

FIG. 8 is a diagram illustrating the modeling of the electric field near the microscope probe tip in an embodiment of the present invention.

FIG. 9 is a flow chart illustrating a method for determining dielectric properties according to the present invention.

25 FIG. 10 is a flow chart depicting an embodiment of a calibration routine of the present invention.

AMENDED SHEET

FIG. 11 is a flow chart depicting an embodiment of a scanning routine of the present invention.

FIG. 12 is a flow chart depicting an embodiment of a probe calibration curve generating routine.

FIG. 13 is a diagram representative of quantitative modeling curves generated in an embodiment of the present invention.

FIG. 14 is a diagram representative of a calibration curve generated in an embodiment of the present invention.

FIG. 15 illustrates the use of a modulating bias voltage to measure dielectric nonlinearity according to an embodiment of the present invention.

FIG. 16 illustrates a probe tip geometry descriptor according to an embodiment of the present invention.

FIG. 17 is a diagram representative of a calibration curve generated in an embodiment of the present invention.

FIG. 18 illustrates frequency shift measurements taken at different heights for several dielectric samples according to an embodiment of the present invention.

FIG. 19 is a diagram representative of calibration curves generated for frequency shift measurements taken at heights of 100 μm and 10 μm according to an embodiment of the present invention.

FIG. 20 illustrates the dielectric constant of a LaAlO_3 sample imaged at 100 μm and 9.08 GHz using a 480 μm diameter probe according to an embodiment of the present invention.

FIG. 21 is a chart displaying dielectric constant values taken from literature and the dielectric constant values obtained from experimental measurements taken according to a method of the present invention.

FIG. 22a illustrates a dielectric constant image of a test sample taken with a 480 μm probe at 9.08 GHz and 100 μm above the sample according to an embodiment of the present invention.

FIG. 22b illustrates the topography of a test sample found according to a method of the present invention.

Detailed Description of the Preferred Embodiments

5 The invention described herein is a system, apparatus, and method for displaying the dielectric properties of bulk and thin film samples. The invention uses a near-field scanning microwave microscope (NSMM).

Physical Design for the System of the Present Invention

10 An apparatus according to an embodiment of the present invention is illustrated in FIG. 1. System 100 shows a near-field scanning microwave microscope having an open-ended coaxial probe 130 with a sharp, protruding center conductor 185. A coaxial transmission line resonator 135 is used for producing a resonance between the probe tip 185 and the capacitive coupler 145. The microwave signal source 165 is responsible for generating a microwave signal. The feedback circuit 160 receives a reflected microwave signal from the coaxial transmission line resonator 135. Feedback circuit 160 also keeps the microwave signal source 165 locked onto a predetermined resonance. An additional function of the feedback circuit 160 is to calculate a value for at least one parameter related to a change in the resonance due to the dielectric properties of a sample 125. Two possible parameters measured are resonant frequency shift (Δf) and quality factor (Q). A stage 120 is used to support the sample 125 in contact with the sharp protruding center conductor 185. Alternatively, with respect to FIG. 2 described in more detail below, a further embodiment of the present invention has a spring-loaded cantilever sample holder 210 provided to hold the sample relative to the sharp protruding center conductor 185 and stage 120.

15

20

25

System 100 further includes motor controllers 115 for manipulating the sample 125 in contact with the sharp protruding center conductor 185 in a first, second, or third direction; for example, the sample can be moved and/or rotated along the x, y and z axes. A coupler 150 is attached between the microwave signal source 165 and the coaxial transmission line resonator 135. Coupler 150 could be a directional coupler, circulator, or other device for directing microwave signals known to one of ordinary skill in the art. The coupler initially directs the microwave signal towards the sample 125 and then directs the reflected microwave signal towards the feedback circuit 160.

A detector 155 is responsible for converting the reflected signal directed towards the feedback circuit 160 into a voltage signal representative of detected power. A computer 105 having both memory and a processor is also shown. The memory of computer 105 stores electric field configuration data files used for quantitative modeling. Computer 105 also stores calibration sample frequency shift values and test sample frequency shift values. The processor of computer 105 mathematically determines the functional relationship between at least one parameter related to a change in the resonant frequency shift and a known dielectric property value of a sample responsible for the change. An example of such a parameter is relative dielectric permittivity (ϵ_r). The processor of computer 105 is then able to receive from feedback circuit 160 resonant frequency shift values for a sample with unknown dielectric properties and determine the value of the unknown dielectric property based upon the model frequency shift-dielectric property relationship. Display device 110 is provided for displaying the value of the dielectric property once it has been determined by the processor 105.

FIG. 2 illustrates an alternative embodiment for the present invention where a spring-loaded sample support 200 is used to support the sample 125 beneath the probe tip 185. Sample support 200 uses a cantilever sample holder 210 having an angled end and a planar surface for supporting sample 125. A first bracing device 230 is fixed to the x-y stage and is joined to sample holder 210 by

5 a spring 220. The spring 220 is positioned such that it is in close proximity to the pivot point created when the angled portion of sample holder 210 is brought into contact with a second bracing device 240 also attached to the x-y stage. This spring cantilever design allows the force applied by the probe tip 185 to remain substantially constant during scanning. Excessive wear of the probe tip during contact mode scanning is also reduced. The amount of force applied can be set by selecting an appropriate spring and adjusting the location of the spring and/or sample along the cantilever sample holder 210. In one example, a force of about 50 μN (microNewtons) is applied between the probe tip and sample 125.

10 System 100 also has a quantitative modeler described in FIG. 3 for determining the model frequency shift-dielectric property relationship. Quantitative modeler 300 receives resonant frequency shift values for calibration samples with known dielectric properties and test samples with unknown dielectric properties from computer 105 memory. Quantitative modeler 300 provides the measured resonant frequency shift data to Model calibration curve generator 310 Probe calibration curve generator 320 and Test sample curve generator 330 in order to generate data (such as, tables, graphs, files or curves) showing the relationship between a measured resonant frequency shift and a particular dielectric property. In particular, Model calibration curve generator 20 310 generates model calibration curves at different values of a geometric descriptor that show the relationship between a measured resonant frequency shift (Δf) and a dielectric property (e.g. permittivity ϵ_r) for calibration reference samples. The operation of Model calibration curve generator 310 is further described with reference to figures 12 and 13. Probe calibration curve generator 25 320 and Test sample calibration curve generator 330 generate calibration curves drawn from the model calibration curves and a geometric descriptor value of the probe tip 185. The calibration curves show the relationship between a resonant frequency shift (Δf) measured during scanning and a dielectric property (e.g. permittivity ϵ_r) at a geometric descriptor value of the actual NSMM used in

5 imaging. The calibration curve allows a dielectric property to be determined by finding the point on the curve corresponding to the resonant frequency shift measured during scanning. The operation of Probe calibration curve generator 320 and Test calibration curve generator 330 is further described with respect to figures 9, 12, 14 and 17.

It is helpful to begin with discussion of quantitative modeling according to the present invention with reference to figures 4 to 8, and 16. Operation of the present invention is then further described with respect to figures 9-15.

Quantitative Modeling

Calculating the Electric Field Near the Probe Tip

10 In order to arrive at quantitative results, the inventors developed a physical model for the system, starting with the simplest case of a uniform bulk sample. Because the probe tip length is much smaller than the free space wavelength of the microwaves (~ 4 cm), a static calculation of the microwave electric fields is performed. Cylindrical symmetry further simplifies the problem to two dimensions. Because of the complicated geometry of the probe tip in contact with a potentially multi-layered sample, a finite element model on a grid is used. Using relaxation potential Φ in the region represented by the grid and taking into account any changes in the permittivity ϵ_r , yields:

20
$$\nabla^2 \Phi + \frac{1}{\epsilon_r} (\nabla \Phi) \cdot (\nabla \epsilon_r) = 0 \quad (1)$$

Using a rectangular grid, the inventors represent the probe tip as a cone with a blunt end of radius r_0 , and perform the calculation in a spreadsheet program.

-10-

From Eq. (1), if Φ_{ij} is the potential at the cell at column i and row j , Φ_{ij} is a weighted average of the values of the four adjacent cells:

$$\Phi_{ij} = \frac{\Phi_{i+1,j} \left(1 + \frac{\Delta r}{r}\right) + \Phi_{i-1,j} + \Phi_{i,j-1} + \Phi_{i,j+1}}{4 + \frac{\Delta r}{r}}, \quad (2)$$

where r is the radius from the cylindrical coordinate axis, referring to FIG. 16, Δr and Δz are the spacing between cells in the r and z directions, respectively, and $\Delta r = \Delta z$. Equation (2) is the simplest case; in practice, the equation is more complicated, such as at the interface between the dielectric sample ($\epsilon_r > 1$) and the air ($\epsilon_r = 1$). Near the probe tip where the electric field is the strongest, and hence, the most critical, the grid spacings Δr and Δz are small and uniform. Inside this box, $\Delta z = \Delta z_{in} = 0.1 \mu\text{m}$, is fixed while the value of $\Delta r = \Delta r_{in}$ is uniform, but adjustable ($0.5 \mu\text{m} \leq \Delta r_{in} \leq 1.0 \mu\text{m}$), to allow for probe tips with different sharpness. In FIG. 16, the geometry descriptor of a probe tip's sharpness is represented with an aspect ratio parameter $\alpha \equiv \Delta z_{in}/\Delta r_{in}$.

The boundaries of the grid should be sufficiently far away in order to minimize the effect of the chosen boundary conditions on the electric field near the probe tip. To accomplish this, outside a region close to the probe tip, the values of Δr and Δz continuously increase with distance away from the probe tip, allowing the outer radius of the grid to be at least 4 mm, and the height of the grid to be 2 mm. The resulting grid consists of 84×117 cells, which is small enough to be a manageable calculation with a modern personal computer. The top and outer boundary conditions are $d\phi/dn = 0$ where n is the coordinate normal to the edge. At the bottom of the grid, which represents the bottom side of the $500 \mu\text{m}$ thick sample, $\phi = 0$ is used for the boundary condition. To match this condition, the sample is placed on top of a metallic layer for scanning; this has the added

AMENDED SHEET

benefit of shielding the microscope from the effects of whatever is beneath the sample, which could be difficult to model.

Two possible fitting parameters for the model are the geometry descriptor, i.e., aspect ratio α , and the radius r_0 of the blunt probe end. The inventors found that a satisfactory fit with data could be obtained by fixing $r_0 = (0.6 \mu\text{m})/\alpha$. This leaves α as a single fitting parameter to represent all probes; the inventors have found that typically $1 < \alpha < 2$. When α is small, for example less than 1.5, the probe tip is considered to be blunt. When α is large, for example greater than 1.5, the probe tip is considered to be sharp.

Calculating the Frequency Shift of the Microscope

Using perturbation theory, the frequency shift of the microscope is calculated as a function of the fields near the probe tip. FIG. 4 illustrates the frequency shift during an unperturbed state, i.e., no sample present and a perturbed state, i.e., sample present beneath the probe. The width of the minima change with respect to the changes in quality factor (Q). With reference to FIG. 5, ϵ_{r1} and ϵ_{r2} are defined as the permittivities of two model samples, the subscripts 1 and 2 indicating the unperturbed and perturbed system. If \vec{E}_1 and \vec{E}_2 are the calculated electric fields inside the two model samples, where $\vec{E} = -\nabla\Phi$, the frequency shift of the microscope upon going from sample 1 to sample 2 is

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV \quad (3)$$

where $\epsilon_0 = 8.85 \times 10^{-12} \text{ F/m}$ is the permittivity of free space, W is the energy stored in the resonator, and the integral is over the volume V_s of the sample. An approximate W is calculated using the equation for the loaded Q of the resonator,

-12-

$Q_L = \omega_0 W/P_l$, where ω_0 is the resonant frequency, and P_l is the power loss in the resonator. In one example, a bare 500 μm thick LaAlO_3 (LAO) substrate is used for the unperturbed system because its properties are well-characterized ($\epsilon_r = 24$) and it is a common substrate for oxide dielectric thin films.

FIG. 6 illustrates the electric fields 605 present when a bulk sample 610 is scanned beneath probe tip 185.

Modeling of Dielectric Thin Films

FIG. 7 illustrates the modeling of thin films. To provide for imaging of thin films, the finite element model is extended to include a thin film 710 on top of a dielectric substrate 715 having the same thickness as the model samples. As long as the thin film 710 is on a substrate having the same thickness as the model sample, the only perturbation is the thin film (the change in total thickness of the sample is negligible compared to the thick substrate). The electric field 705 in the thin film sample is calculated using the finite element model of the present invention and Eq. (3) to calculate Δf , integrating only over the volume of the thin film. Once a probe's α parameter is found using the process described above for bulk samples, the thin film model is used to obtain a functional relationship between Δf and ϵ_r of the thin film.

In producing the quantitative models discussed above, samples are chosen so as to cover a range of permittivity values in which the dielectric permittivity of an unknown sample is expected to fall within. The known values ϵ_r and t (thickness of the thin film 710) and the calculated values α and Φ are all stored in electric field configuration data files respective to each sample. In FIG. 8 for example, quantitative modeling data has been generated using samples with dielectric permittivity values of 10 to 200 and probe tip geometry of $\alpha=1$ and $\alpha=2$. Accordingly, the electric field configuration data files are usable in cases where

AMENDED SHEET.

ART 34 AMDT

-13-

the dielectric permittivity for an unknown sample can be expected to be between 10 and 200.

The following articles are related to quantitative modeling according to the present invention and are incorporated by reference herein in their entirety:

5 "Imaging of microwave permittivity, tunability, and damage recovery in (Ba, Sr) TiO₃ thin films", D.E. Steinhauer, C.P. Vlahacos, F.C. Wellstood, Steven M. Anlage, C. Canedy, R. Ramesh, A. Stanishevsky, and J. Melngailis, *Applied Physics Letters*, Volume 75, Number 20, p. 3180 (1999); and

10 "Non-Contact Imaging of Dielectric Constant with a Near-Field Scanning Microwave Microscope", C.P. Vlahacos, D. E. Steinhauer, Steven M. Anlage, F.C. Wellstood, Sudeep K. Dutta, and Johan B. Feenstra, *The Americas Microscopy and Analysis*, p. 5, January 2000.

Method of the Present Invention

Dielectric Permittivity Imaging

15 A method for an embodiment of the present invention is illustrated by flowchart 900 of FIG. 9. The method begins with a step 905. In a step 910, system 100 is calibrated according to the probe calibration routine described in FIG. 10 to obtain calibration sample frequency shift values ($\Delta f_{\text{sampleC}}$), a probe tip geometry descriptor, and sample geometry.

Test Sample Scanning

20 In a step 915, a test sample 125 with unknown dielectric properties is scanned according to the routine described in FIG. 11 to obtain test sample resonant frequency shift measurements ($\Delta f_{\text{sampleT}}$). These measurements can also include determining quality factor (Q), and/or tunability information. If the test

AMENDED SHEET

ART 34 AMDT

-14-

sample is a thin film then it contains both a dielectric thin film and substrate. Step 915 will now be described with reference to the steps provided in FIG. 11. The method begins with a step 1105. In a step 1110 a resonant frequency of the near-field scanning microwave microscope is selected. In a step 1115 a test sample is placed on the microscope stage. In a step 1120 the probe tip is moved into contact with the test sample. In a step 1125 raster scanning begins. In a step 1130 a position value corresponding to the initial point of contact with the test sample is stored in computer 105 memory as the next scanning point.

In a step 1135 the probe is moved to a predetermined background measurement position where the calibration sample no longer perturbs the resonator. For example, the predetermined background measurement position can be a height 1.5 times greater than the diameter of the microscope transmission line 135, or a height at least approximately 3 millimeters above the calibration sample. In a step 1140 a background resonant frequency shift measurement is taken from the background measurement position. In a step 1145 the probe tip is moved back into contact with the test sample at the next scanning point. In a step 1150 the contact resonant frequency shift at the scanning point is measured. In a step 1155 the difference between the contact resonant frequency shift at the scanning point and the background resonant frequency shift is calculated. In a step 1160 the calculated difference is saved in computer 105 memory as the test sample resonant frequency shift value.

In a step 1165 the microscope probe tip is moved to the next scanning position. In a step 1170 computer 105 determines if the next scanning position is the end of a scan line. If the end of a scan line has not been reached then steps 1150 through 1165 are repeated. Next, at a step 1175 computer 105 determines if the next scanning position is the end of a scan area. If it is not then steps 1135 through 1170 are repeated until the end of a scan area has been reached. At the conclusion of step 915 a set of test sample resonant frequency values have been recorded or stored in computer 105 memory.

AMENDED SHEET

5 In a step 920, the calibration sample resonant frequency shift values
obtained from step 910 are used to generate a probe calibration curve according
to the routine described in FIG. 12. In a step 925, a determination of whether the
test sample is a thin film is made. In a step 930, a determination of whether all
10 of the calibration samples have the same thickness as the test sample is made.
If the test sample is not a thin film, and all of the calibration samples have the
same thickness as the test sample, then in a step 945, one is able to determine an
unknown dielectric property for a sample by first retrieving a test sample resonant
frequency shift value ($\Delta f_{\text{sampleT}}$) obtained in step 915 and then locating the
15 corresponding Δf value on the probe calibration curve resulting from step 920.
The dielectric property of the test sample is equal to the dielectric property
corresponding to the Δf value on the probe calibration curve resulting from step
920. If the test sample is a thin film, or if only one calibration sample has the
same thickness as the test sample, then in a step 935 a test sample calibration
20 curve is generated. In a step 940 one is able to determine an unknown dielectric
property for a test sample by first retrieving a test sample resonant frequency
shift value ($\Delta f_{\text{sampleT}}$) obtained in step 915 and then locating the corresponding Δf
value on the test sample calibration curve resulting from step 940. The dielectric
property of the test sample is equal to the dielectric property corresponding to the
25 Δf value on the test sample calibration curve. The process concludes with a step
950.

In an alternative embodiment, step 920 can be performed prior to step
915.

25 In a further embodiment, step 910 can be performed after step 915 but before step
920. Furthermore, steps 1135, 1140, and 1145 could alternatively be measured
at each point on the sample, any desired number of times during the scan, before
the scan, or after the scan.

AMENDED SHEET

Calibration

To measure the unknown dielectric permittivity (ϵ_r) of a dielectric sample, the NSMM is calibrated to determine the parameter α using at least two samples with known permittivity (ϵ_r) and thickness (t). The thickness of a bulk dielectric test sample or a thin film test sample on top of a dielectric substrate, is referred to as the first determined thickness. For thin film dielectric test samples the thickness of the thin film is referred to as the second determined thickness.

The process of calibrating system 100 is illustrated by flowchart 1000 of FIG. 10. The process starts at a step 1010. At a step 1015, at least two calibration samples that have known dielectric properties are selected. At least two of the calibration samples have the same approximate thickness with one another. In addition, at least one of the calibration samples has the same approximate thickness as the test sample with first determined thickness and unknown dielectric properties. If the test sample contains a thin film, an additional requirement is that the at least one calibration sample with approximate thickness equal to the test sample first determined thickness have the same permittivity as the substrate of the test sample.

Calibration Sample Scanning

In a step 1020, each selected calibration sample is scanned according to the scanning routine described in FIG. 11 to determine respective sets of resonant frequency shift information for samples with known dielectric permittivity values. The method begins with a step 1105. In a step 1110 a resonant frequency of the near-field scanning microwave microscope is selected. In a step 1115 a calibration sample is placed on the microscope stage. In a step 1120 the probe tip is moved into contact with the calibration sample. In a step 1125 raster scanning begins. In a step 1130 a position value corresponding to the initial point of

ART 34 AMDT

-17-

contact with the calibration sample is stored in computer 105 memory as the next scanning point. In a step 1135 the probe is moved to a predetermined background measurement position where the calibration sample no longer perturbs the resonator. For example, the predetermined background measurement position can be a height 1.5 times greater than the diameter of the microscope transmission line 135, or a height at least approximately 3 millimeters above the calibration sample. In a step 1140 a background resonant frequency shift measurement is taken from the background measurement position. In a step 1145 the probe tip is moved back into contact with the calibration sample at the next scanning point. In a step 1150 the contact resonant frequency shift at the scanning point is measured. In a step 1155 the difference between the contact resonant frequency shift at the scanning point and the background resonant frequency shift is calculated. In a step 1160 the calculated difference is saved in computer 105 memory as the calibration sample resonant frequency shift value. In a step 1165 the microscope probe tip is moved to the next scanning position. In a step 1170 computer 105 determines if the next scanning position is the end of a scan line. If the end of a scan line has not been reached then steps 1150 through 1165 are repeated. Next, at a step 1175 computer 105 determines if the next scanning position is the end of a scan area. If it is not then steps 1135 through 1170 are repeated until the end of a scan area has been reached. Step 1020 is repeated for each calibration sample.

At a step 1025, a geometry descriptor of the probe tip is determined. The geometry descriptor can be input by a user or calculated by System 100. A geometry descriptor can be any descriptor representative of the geometry of the probe tip. Accordingly, in one embodiment of the present invention, a geometry descriptor is referred to as a first probe tip geometry descriptor value and a second probe tip geometry descriptor value. In another embodiment, an aspect ratio $\alpha = \Delta z / \Delta r$ is used where, Δz is a distance along a z-direction parallel to a length of the

APPROVED SHEET

resonator and the probe tip and Δr is a radius distance extending from the central axis of the probe tip 185 to its outermost surface.

At a step 1030, sample geometry data is input. For example, with bulk samples, sample geometry data includes the thickness of the sample. For thin film samples on a bulk substrate, the thickness of the thin film is provided as well.

Calibration Curve Generation

FIG 12 describes the routine for generating a calibration curve. The routine begins at a step 1205. At a step 1210 electric field configuration data is stored as described in FIG 8. The stored files contain data for model samples having approximately the same thickness as the calibration sample scanned in step 1020. At least one file for each value of α has the same permittivity value as one of the calibration samples scanned in step 1020. This permittivity is defined to give the unperturbed system, with $\Delta f=0$.

At a step 1215 model calibration curves are generated by reading from at least two of the previously stored electric field configuration data files, electric field values and permittivity values for at least two respective probe tip geometry descriptor values. FIG. 13 provides an example of model calibration curves for $\alpha=1$ and $\alpha=2$. To generate the model calibration curve where $\alpha=1$, a first previously stored electric field value (E_1) and a first permittivity value (ϵ_{r1}) is chosen to represent the zero point ($\Delta f=0$). A second electric field configuration data file where $\alpha=1$ is then read to obtain a second previously stored electrical field value (E_2) and a second permittivity value (ϵ_{r2}). A point on the first model calibration curve is then generated by solving the equation

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV$$

AMENDED SHEET

ART 34 AMDT

-19-

Multiple points on the first model calibration curve can be generated by repeating step 1215 for different values of ϵ_{r2} and E_2 . The second model calibration curve for $\alpha=2$ is generated as described above except that electric field configuration data files where α equals two are used instead.

5 At a step 1220, a calibration curve is generated using the model calibration curves from step 1215 and the calibration sample frequency shift values from step 1020. A point on the calibration curve is generated by first calculating the difference between two calibration sample frequency shift values. One of the calibration frequency shift values used in the calculation should correspond to zero point permittivity value from step 1215. Once the difference value has been determined it can be plotted with respect to the Δf axis of the model calibration curve. FIG. 14 provides an example where four calibration points have been plotted with respect to the model calibration curve of FIG. 13 to produce a calibration curve for $\alpha=1.5$. The value for α for the probe is determined by observing the position of the curve C_p relative to the curves C_{m2} and C_{m1} .

Test Sample Calibration Curve Generation

15 If the test sample is a thin film sample, or if only one calibration sample has the same thickness as the first determined thickness of the test sample, then a separate test sample calibration curve must be generated, in step 935.

20 An example test sample calibration curve C_{ts} is shown in FIG. 17. The curve C_{ts} is used in step 940 to convert the frequency shift values obtained in step 915 to the values of the permittivity of the test sample. The curve C_{ts} can be calculated using one of two methods. For the first method, the calculation is done for the specific value of α determined in step 920 (such as $\alpha = 1.5$, as shown in FIG. 17). For the second method, the calculation is done for two or more values of α , shown in FIG. 17 as the curves C_{ts1} and C_{ts2} . Then, the curve C_{ts} is

AMENDED SHEET

ART 34 AMDT

-20-

calculated for the value of α determined in step 920 by interpolating between the curves C_{ts1} and C_{ts2} . The advantage of the second method is that generating the curves C_{ts1} and C_{ts2} can be done in advance just once, requiring less calculation for each scan.

5 For the case where the test sample is a bulk sample, and only one calibration sample has the same thickness as the test sample, the curves in FIG 17 are calculated using the same method as was performed in the previous section with the probe calibration curve. The difference is that a new set of files (represented in FIG. 8) are used, which represent a sample thickness equal to the test sample thickness. The frequency shift measured with the NSMM for the calibration sample having the same thickness as the test sample (done in step 910) is defined as $\Delta f=0$ for the test sample calibration curves in FIG. 17.

10 For the case of a thin film test sample, the curves C_{ts1} and C_{ts2} in FIG. 17 are calculated according to the routine described in FIG. 12. However, a different set of files represented in FIG. 8 is now used. In this new set, the files represent a thin film having a thickness equal to the second determined thickness of the thin film in the test sample. The volume V_s is the volume of the thin film, rather than the volume of the entire sample. The frequency shift measured with the NSMM for the calibration sample which has the same thickness as the test sample, and the sample permittivity as the test sample substrate, is defined as $\Delta f = 0$ for the curves in FIG. 17.

Nonlinear Dielectric Imaging

25 FIG. 15 illustrates how electric field-dependent imaging can be accomplished by applying a voltage bias (V_b) 1505 to the probe tip via a bias tee 180 in the resonator according to a further feature of the present invention. A metallic layer 1520 beneath the thin film 1510 acts as a grounded counterelectrode.

AMENDED SHEET

As shown in FIG 15, a sample 125 can be a dielectric thin film 1510. The dielectric thin film 1510 is disposed on a grounded counterelectrode 1520. The grounded counterelectrode 1520 is provided on a substrate 1530. In order to prevent the counterelectrode 1520 from dominating the microwave measurement (modeling of the system has shown this to be a potential problem), the inventors use a high-sheet-resistance counterelectrode, making it virtually invisible to the microwave fields. As a result, the presence of the thin-film counterelectrode 1520 can be safely ignored in the finite element model described above. Because the counterelectrode 1520 is immediately beneath the dielectric thin film, the applied electric field is primarily in the vertical direction, unlike the microwave electric field, which is mainly in the horizontal direction for thin films with large permittivities. Also, by simulating the applied field using a finite element model similar to that presented above, the inventors find that the applied electric field beneath the probe tip is approximately uniform and equal to $E_b = V_b/t_f$, where t_f is the thickness of the dielectric thin film.

By modulating the bias voltage 1505 applied to the probe tip, nonlinear terms in the permittivity can be extracted. Expanding the electric displacement D in powers of the electric field E , and keeping only the nonzero terms yields

$$D_1(E) = \epsilon_{11}E_1 + \frac{1}{2}\epsilon_{113}E_1E_3 + \frac{1}{6}\epsilon_{1133}E_1E_3^2 + \dots \quad (4)$$

where E_1 is the rf electric field in the r direction, and $E_3 = E_b$ is the applied bias electric field in the z direction.

Adding a low-frequency oscillatory component ($\omega_b = 1$ kHz, with an amplitude of $\tilde{V}_b = 1$ V, for example) to the bias voltage, the applied electric field is $E_b = E_b^{dc} + \tilde{E}_b \cos \omega_b t$. The effective rf permittivity is then

AMENDED SHEET

-22-

$$\begin{aligned}
 \varepsilon_{rf} = & \varepsilon_{11} + \varepsilon_{113} E_b^{dc} + \varepsilon_{1133} \left(\frac{(E_b^{dc})^2}{2} + \frac{\tilde{E}_b}{4} \right) \\
 & + (\varepsilon_{113} + \varepsilon_{1133} E_b^{dc}) \tilde{E}_b \cos(\omega_b t) \\
 & + \frac{1}{4} \varepsilon_{1133} \tilde{E}_b^2 \cos(2\omega_b t) + \dots
 \end{aligned} \tag{5}$$

Note that the components of ε_{rf} at ω_b and $2\omega_b$ are approximately proportional to ε_{113} and ε_{1133} , respectively. Expanding the resonant frequency of the microscope as a Taylor series about $f_0(\varepsilon_{rf} = \varepsilon_{11})$, yields

$$f_0[\varepsilon_{rf}(t)] = f_0(\varepsilon_{11}) + \left. \frac{df_0}{d\varepsilon_{rf}} \right|_{\varepsilon_{rf}=\varepsilon_{11}} [\varepsilon_{rf}(t) - \varepsilon_{11}] + \dots \tag{6}$$

Substituting Eq. 5 into Eq. 6, and keeping only the larger terms,

$$\begin{aligned}
 f_0(t) \approx & \text{constant} + \varepsilon_{113} \tilde{E}_b \left. \frac{df_0}{d\varepsilon_{rf}} \right|_{\varepsilon_{11}} \cos(\omega t) \\
 & + \frac{1}{4} \varepsilon_{1133} \tilde{E}_b^2 \left. \frac{df_0}{d\varepsilon_{rf}} \right|_{\varepsilon_{11}} \cos(2\omega t).
 \end{aligned} \tag{7}$$

Thus, the components of the frequency shift signal at ω_b and $2\omega_b$ can be extracted to determine the nonlinear permittivity terms ε_{113} and ε_{1133} . These nonlinear terms can be measured simultaneously with the linear permittivity (ε_{11}) while scanning.

APPENDED SHEET,

ART 34 AMDT

-23-

As an alternative, the electric field E_x could be applied in the horizontal direction using thin film electrodes deposited on top of the dielectric thin film. The advantage here is that diagonal nonlinear permittivity tensor terms could be measured, such as ϵ_{111} and ϵ_{1111} ; the disadvantage in this case is that imaging is limited to the small gap between the electrodes.

Simultaneously Measuring Sample Topography

Using a spring-loaded sample holder such as that shown in FIG. 2, one can also measure sample topography. This is accomplished by measuring the deflection of the sample holder during a scan. Because the probe tip is held fixed, the sample holder will deflect depending on the topography of the sample. For example, if the sample has a bump on top of it, the sample holder will deflect downward. One can record this deflection during a scan, and obtain a topographical image corresponding to the same region as the microwave image(s).

The measurement of the deflection of the sample holder could be accomplished using one of many techniques, including an optical sensor, a capacitive sensor, an interferometer, etc.

Overview

The microwave microscope, as covered in patent 5,900,618, consists of a resonator contained in a microwave transmission line. One end of the resonator is an open-ended coaxial probe, which is held close to the sample, and the other end is coupled to a microwave source with a coupling capacitor. A sample is scanned beneath the probe. Because of the concentration of the microwave fields at the probe center conductor, the resonant frequency and quality factor Q of the resonator are perturbed depending on the properties of the sample near the probe

AMENDED SHEET

center conductor. One quantity recorded while scanning is the resonant frequency shift (Δf) of the resonator.

Two modes of operation are non-contact mode and contact mode. In non-contact mode, the preferred embodiment is with the probe center conductor flush with the face of the probe, so that the end of the center conductor is in the same plane as the end of the outer conductor. A sample is scanned beneath the probe with a small gap of 10-100 μm between the probe and the sample. In contact mode imaging, the center conductor extends beyond the outer conductor, and has a sharp point. The sample holder gently presses the sample against the probe tip with a small force.

The invention involves calibration using dielectric samples with known permittivity. In the case of non-contact mode, the calibration data is interpolated, allowing one to scan dielectric samples with the same thickness as the calibration samples, and to convert the microscope frequency shift into the local permittivity of the sample. In the case of contact-mode imaging, a physical model is used to generate the relationship between the frequency shift of the microscope and the local permittivity of the sample. Because of the use of the model with contact-mode imaging, quantitative imaging in this case is not limited to samples which have the same thickness as the calibration samples. The contact-mode imaging can be used for both bulk and thin-film samples.

In contact mode, a low-frequency electric field can also be applied to the sample, so that permittivity can be measured as a function of the applied electric field. Thus, dielectric nonlinearity can be measured as well as the linear permittivity.

When scanning in contact mode, an optical sensor can be used to measure the deflection of the sample holder, and hence, the sample, as the sample is scanned in contact with the probe tip. The deflection is exactly equal to the topographic changes in the sample. Thus, by recording this reflection at the same

AMENDED SHEET

time that Δf is measured, the sample topography can be imaged simultaneously with the permittivity.

In one embodiment of non-contact mode imaging, the local dielectric constant of a material is determined by measuring the frequency shift of the microscope as a function of height above the sample. Areas as small as 100 μm in diameter can be measured.

An unknown sample is scanned and a dielectric constant is measured as a function of position as long as the height of the probe above the sample is accurately known.

The technique can be performed quickly and at many locations on a bulk dielectric material.

The techniques can be done over a very broad range of frequencies simply by choosing other resonant modes of the microscope. In principle one can measure between about 100 MHz and 50 GHz.

The technique can be applied over a broad range of temperatures, from 1.2K to well above room temperature, possibly as high as 1000°C.

Non-Contact Imaging of Dielectric Constant with a Near-Field Scanning Microwave Microscope

In another embodiment of the present invention, a non-contact technique for imaging dielectric constant using a resonant near-field scanning microwave microscope is provided. By measuring the shift in the system's resonant frequency during a scan over an insulating sample, one can obtain quantitative images of dielectric variations. In one example, the inventors scanned seven samples with dielectric constants ϵ_r ranging from 1 to 230, using a 480 μm diameter probe at a height of 100 μm and a frequency of 9.08 GHz. The technique achieves an accuracy of about 25% for $\epsilon_r=230$ and less than 2% for $\epsilon_r=2.1$, limited mainly by variations in the probe-sample separation.

AMENDED SHEET

This approach offers a fast, simple, broadband method to image dielectrics using readily available microwave components.

In one embodiment, resonant near-field scanning microwave microscope consists of a 1m long coaxial transmission line which is capacitatively coupled to a microwave source at one end and terminated by an open-ended coaxial probe at the other end. This arrangement creates a resonant circuit in which the resonant frequency f_0 and quality factor Q are modified when a sample approaches the open end of the probe (see inset in Fig. 18). By using a frequency-following feedback circuit the invention keeps the microscope source locked on resonance. The shift of the system's resonant frequency Δf as the sample under the probe is scanned. The variations in Δf are directly related to spatial variations in dielectric constant in the sample. In addition, however, topographic changes will also give rise to changes in Δf .

Calibration Example

To calibrate the system, the inventors constructed a test sample by placing six pieces of different dielectric material into the bottom of a square plastic mould and pouring epoxy into the mould. In addition, silicone adhesive was used to hold each piece down. After the epoxy cured, the test sample was removed from the mould, polished, and positioned on the XY table. The materials embedded in the epoxy were silicon, glass microscope slide, SrTiO_3 , Teflon, sapphire, and LaAlO_3 . All six pieces were approximately $500\text{ }\mu\text{m}$ thick and about $6\text{ mm} \times 8\text{ mm}$ in size. The overall thickness of the test sample was 6 mm .

The frequency shift Δf versus height h above the six pieces having dielectric constants ranging from 2.1 to about 230 is measured. The inventors also tested the epoxy which has an unknown dielectric constant. Each piece, as well as the probe, was flat and smooth on the scale of $5\text{ }\mu\text{m}$ as judged by an optical microscope. For these measurements, a probe with a $480\text{ }\mu\text{m}$ center

AMENDED SHEET

ART 34 AMDT

-27-

conductor diameter and a source frequency of 9.08 GHz was used in one example of implementation. For each scan, the probe was first brought in contact with a dielectric sample and the frequency shift Δf was recorded as the height was systematically increased. The results are plotted in Fig. 18. Samples with the largest dielectric constant produced the largest frequency shift, as expected. The largest shift observed was -26.2 MHz, when the probe was in contact with a SrTiO₃ sample with $\epsilon_r=230$. The smallest shift found was -1.2 MHz when the probe was in contact with a Teflon sample with $\epsilon_r=2.1$. As can be seen from Fig. 18, the frequency shift is essentially zero above 1 mm and saturates when the probe-sample distance is smaller than a few microns.

The inventors used the above information to construct an empirical calibration curve that directly relates the frequency shift to the dielectric constant ϵ_r . In order to construct the calibration curve the inventors took the difference between the frequency shift at two different heights h_1 and h_2 , i.e., $f_d = \Delta f(h_2) - \Delta f(h_1)$, where h_2 is far away ($h_2 > 1000 \mu\text{m}$). By taking the difference, the effect of drift in the microwave source frequency is eliminated. As shown in Fig. 19, two calibration curves of f_d versus ϵ_r can be constructed, one curve for $h_1=10 \mu\text{m}$ and $h_2=1.1 \text{ mm}$ and the other for $h_1=100 \mu\text{m}$ and $h_2=1.1 \mu\text{m}$. The parameters in each calibration curve are set with an empirical function (solid lines in Fig. 19), allowing us to easily transform any measured frequency shift to a dielectric constant. From these curves one can see that one can enhance the sensitivity to the dielectric constant considerably by using a small probe height. On the other hand, at closer probe-sample separations the influence of topographic features will be enhanced.

AMENDED SHEET

ART 34 AMDT

-28-

Imaging Results

To test the dielectric imaging capabilities of our system, the inventors next scanned a single sample of LaAlO_3 which had an 8 x 5 mm triangular shape and a thickness of 510 μm . The inventors placed the sample directly on the metal scanning table and recorded the frequency shift as a function of position. The data was taken at 9.08 GHz using the 480 μm probe at heights of 100 μm and 1.1 mm. The two data sets are subtracted and a 100 μm calibration curve is used to transform the resulting frequency shift image into a dielectric constant image. Figure 20 shows the resulting contour image of dielectric constant versus position. The dielectric constant varies from about 20 to 25 over the sample and equals 1 when the probe is away from the sample. For comparison, the reported value of relative permittivity for LaAlO_3 at room temperature is $\epsilon_r=23.9$ at 18 GHz. In this image, the main variation in ϵ_r over the sample is due to a slight tilt in the sample surface of about 20 μm . The edges of the sample show a smaller value of ϵ_r due to averaging over the inner conductor of the probe. The width of the affected region is in good agreement with the expected spatial resolution of about 500 μm .

The inventors next recorded a frequency shift image of the six-piece test sample using a probe-sample separation of 100 μm and the 480 μm diameter probe at 9.08 GHz. As before, using the $\epsilon_r(f_d)$ calibration, the frequency shift image is transformed into a dielectric constant image (see Fig. 22a). The darker regions in Fig. 22a indicate a higher dielectric constant (larger frequency shift) and the lighter regions indicate a smaller dielectric constant (smaller frequency shift). Figure 22b shows the corresponding surface plot representation. Note that the z-axis in Fig. 22b uses a logarithmic scale to allow us to show the large range of dielectric constants present in the sample. As expected, the largest dielectric constant materials (SrTiO_3 and LaAlO_3) are the highest surfaces and the smallest dielectric constant materials (Teflon and vacuum) are the lowest. Further, notice

PART 34 AMDT

-29-

that the Teflon sample forms a depression in Fig. 22b, indicating that the dielectric constant of Teflon is lower than the surrounding epoxy. Note that voids in the epoxy can easily be seen as irregularly shaped low-dielectric regions (white regions in Fig. 22a) in the epoxy. Fig. 21 summarizes the dielectric constants for the six test materials and provides comparative data taken from literary sources.

From Fig. 22b, it is apparent that there is some noise in our images of dielectric constant. In our system, the predominant sources of random errors are noise in our recording electronics and fluctuations in the source frequency. To establish the precision to which ϵ_r can be determined, we determined the standard deviation in ϵ_r over a small region near the center of the LaAlO_3 sample in Fig. 20; we found $\Delta \epsilon_r = 0.06$ for a sampling time of 30 ms. Similarly, over the Teflon in Fig. 22a, the standard deviation in ϵ_r was 7×10^{-4} for a sampling time of 30 ms. From Fig. 22 we can also estimate the absolute accuracy of our technique. For $\epsilon_r = 230$ (SrTiO_3) the accuracy is about 25% while for $\epsilon_r = 2.1$ (Teflon) the accuracy is better than 2%. The main source of these errors is topographic variations. In our test sample, even after polishing, there are small height variations of about $30 \mu\text{m}$ (e.g. between the sapphire and SrTiO_3 in Fig. 22) between the different dielectrics. Such height variations cause an additional frequency shift, resulting in an error in the measured dielectric constant. In this regard, an accurate measurement of the dielectric constant of a single flat dielectric sample is considerably easier. However, the image of the composite sample clearly demonstrates the strength and sensitivity of this non-contact technique to measure variations in ϵ_r .

Conclusion

While various embodiments of the present invention have been described above, it should be understood that they have been presented by way of example, and not limitation. It will be apparent to persons skilled in the relevant art(s) that

PROCESSED SHEET

ART 34 AMDT

various changes in form and detail can be made therein without departing from the spirit and scope of the invention. Thus the present invention should not be limited by any of the above-described exemplary embodiments, but should be defined only in accordance with the following claims and their equivalents.

[illegible]

8

1944

PART 34 AMDT

-31-

~~What Is Claimed Is:~~

CLAIMS

1. A method for contact imaging of dielectric permittivity using a near-field scanning microwave microscope having a resonator with a probe tip comprising the steps of:
 - (a) calibrating the near-field scanning microwave microscope to determine a geometry descriptor of the probe tip;
 - (b) generating calibration curves;
 - (c) scanning a test sample in contact with the probe tip at scanning locations and generating at least one test sample frequency shift value at each scanning location; and
 - (d) determining the dielectric permittivity of the test sample at the sample locations based on the respective generated test sample frequency shift values and the generated calibration curves.
2. The method of claim 1, wherein said calibrating step (a) includes the steps of:
 - (i) selecting a resonant frequency of the near-field scanning microwave microscope;
 - (ii) scanning each of said at least two calibration samples, where for each scan the probe tip is first brought into contact with each of said at least two calibration samples;
 - (iii) moving the microscope probe to a predetermined background measurement position;
 - (iv) measuring a background resonant frequency at said predetermined background measurement position;

ART 34 AMDT

-32-

- (v) moving the probe tip into contact with said one of said at least two calibration samples at a scanning position;
- (vi) measuring a contact resonant frequency at said scanning position;
- (vii) calculating the difference between said contact resonant frequency and said background resonant frequency;
- (viii) storing in memory said calculated difference as a calibration sample resonant frequency shift value;
- (ix) moving the sample to the next scanning position;
- (x) determining if said next scanning position is the end of a scan line;
- (xi) repeating steps (vi) through (x) until said end of a scan line has been reached;
- (xii) moving the sample to the next scan line;
- (xiii) determining if said next scan line is the end of a scan area;
- (xiv) repeating steps (iii) through (xiii) for each of said at least two calibration samples until said end of a scan area has been reached.

3. The method of claim 2, wherein the resonator includes a microscope transmission line, and wherein said step (iii) comprises moving the microscope probe to a height 1.5 times greater than the diameter of the microscope transmission line.

AMENDED SHEET

PART 34 AMDT

-33-

4. The method of claim 2, wherein said step (iii) comprises moving the microscope probe to a height where the calibration sample no longer perturbs the resonator.
5. The method of claim 2, wherein said step (iii) comprises moving the microscope probe to a height at least approximately 3 millimeters above the calibration sample.
6. The method of claim 1, wherein the geometry descriptor comprises an aspect ratio of the probe tip, and the calibration step (a) further includes calculating the aspect ratio of the microscope probe tip as $\Delta z/\Delta r$, where Δz is a distance along a z-direction parallel to a length of the resonator and the probe tip and Δr is a radius distance extending from the central axis of the probe tip to its outermost surface.
7. The method of claim 1, wherein the scanning step (c) includes the steps of:
- (i) selecting a resonant frequency of the near-field scanning microwave microscope;
 - (ii) placing said test sample on the microscope stage;
 - (iii) moving the probe tip into contact with said test sample;
 - (iv) storing a position value corresponding to the point of contact with said test sample;
 - (v) moving the probe to a predetermined background measurement position;
 - (vi) measuring a background resonant frequency at said background measurement position;

AMENDED SHEET

ART 34 AMDT

-34-

- (vii) moving the probe tip into contact with said test sample at the scanning position;
- (viii) measuring the contact resonant frequency at said scanning position;
- (ix) calculating the difference between said contact resonant frequency and said background resonant frequency;
- (x) storing in memory said calculated difference as a test sample resonant frequency shift value;
- (xi) moving the sample to the next scanning position;
- (xii) determining if said next scanning position is the end of a scan line;
- (xiii) repeating steps (viii) through (xii) until said end of a scan line has been reached;
- (xiv) moving the sample to the next scan line;
- (xv) determining if said next scan line is the end of a scan area;
- (xvi) repeating steps (v) through (xv) until said end of a scan area has been reached.

8. The method of claim 7, wherein the resonator includes a microscope transmission line, and wherein said step (v) comprises moving the microscope probe to a height that is at least approximately 1.5 times greater than the diameter of the microscope transmission line.

9. The method of claim 7, wherein said step (v) comprises moving the probe to a height where the test sample no longer perturbs the resonator.

AMENDED SHEET

ART 34 AMDT

-35-

10. The method of claim 7, wherein said step (v) comprises moving the probe to a height at least approximately 3 millimeters above the test sample.
11. The method of claim 1, wherein the scanning step (c) includes placing a bulk test sample on the microscope stage.
12. The method of claim 11, wherein the generating a calibration curve step (b) includes the steps of:
- (i) storing electric field configuration data files, wherein the data corresponds to a model sample having approximately the same first thickness and further having approximately the same permittivity as one of said at least two calibration samples with known dielectric properties; and wherein the data stored is representative of electric field values at respective ϵ_r and α over a predetermined range of ϵ_r and α , where ϵ_r is a dielectric permittivity value and α is the probe tip geometry descriptor;
 - (ii) generating a first model calibration curve using said stored electric field configuration data;
 - (iii) generating a second model calibration curve using said stored electric field configuration data;
 - (iv) generating a probe calibration curve using said first and second generated model calibration curves and said generated calibration sample frequency shift values; and

AMENDED SHEET

ART 34 AMDT

-36-

- (v) determining the geometry descriptor of the probe tip at the time of scanning of the test sample based upon the positioning of said generated probe calibration curve in relation to said first and second generated model calibration curves.

13. The method of claim 12 wherein the generating a first model calibration curve using said previously stored electric field configuration data (ii) includes the steps of:

- (1) reading from a first electric field configuration data file where α = a first probe tip geometry descriptor value, a first previously stored electric field value (E_1) and a first permittivity value (ϵ_{r1});
- (2) reading from a second electric field configuration data file where α = a first probe tip geometry descriptor value, a second previously stored electrical field value (E_2);
- (3) calculating a point on the first model calibration curve by solving the equation

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV ; \text{ and}$$

- (4) repeating steps (1) through (3) until a predetermined number of points on the first model calibration curve have been generated.

AMENDED SHEET

ART 34 AMDT

-37-

14. The method of claim 12 wherein the generating a second model calibration curve using said previously stored electric field configuration data files step (iii) includes the steps of:

- (1) reading from a first previously stored electric field configuration data file where α =a second probe tip geometry descriptor value, a first previously stored electric field value (E_1) and a first permittivity value (ϵ_{r1});
- (2) reading from a second previously stored electric field configuration data file where α = a second probe tip geometry descriptor value, a second previously stored electric field value (E_2) and a second permittivity value (ϵ_{r2});
- (3) calculating a point on the second model calibration curve by solving the equation

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV ; \text{ and}$$

- (4) repeating steps (1) through (3) until a predetermined number of points on the second model calibration curve have been generated.

15. The method of claim 1, wherein the scanning step (c) includes arranging a thin film test sample on top of a dielectric substrate having approximately the same first determined thickness and the same permittivity as at least one of said calibration samples and then placing said arrangement on the microscope stage.

APPENDIX C

ART 34 AMDT

-38-

16. The method of claim 15, wherein the generating a calibration curve includes the steps of:

(i) storing electric field configuration data files

wherein the data corresponds to a model sample having a thin film of approximately the same second determined thickness and a known dielectric permittivity value arranged on top of said dielectric substrate having approximately the same first determined thickness and the same permittivity as at least one of said calibration samples with known dielectric properties; wherein the stored data files are representative of electric field values at respective ϵ_r and α over a predetermined range of ϵ_r and α , wherein ϵ_r is a dielectric permittivity value for the model sample thin film and α is representative of a probe tip geometry descriptor;

(ii) generating a first test sample calibration curve using said stored electric field configuration data files;

(iii) generating a second test sample calibration curve using said stored electric field configuration data files;

(iv) generating an additional test sample calibration curve using said first and second test sample calibration curves and said generated test sample frequency shift values;

17. The method of claim 16 wherein the generating a first test sample calibration curve using said previously stored electric field configuration data files step (ii) includes the steps of:

(1) reading from a first electric field configuration data file where α = a first probe tip geometry descriptor value, a first previously stored electric field value (E_1) and a first permittivity value (ϵ_{r1}) where ϵ_{r1} is the permittivity of said thin film having approximately the same second determined thickness and known dielectric permittivity;

(2) reading from a second electric field configuration

APPROVED

PART 34 AMDT

data file where α = a first probe tip geometry descriptor value, a second previously stored electric field value (E_2) and a second permittivity value (ϵ_{r2}) where ϵ_{r2} is the permittivity of said thin film having approximately the same second determined thickness and known dielectric permittivity;

5 (3) calculating a point on the first test sample calibration curve by solving the equation

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV ; \text{ and}$$

10 (4) repeating steps (1) through (3) until a predetermined number of points on the first test sample calibration curve have been generated.

18. The method of claim 16 wherein the generating a second test sample calibration curve using said previously stored electric field configuration data files step (iii) includes the steps of:

15 (1) reading from a first previously stored electric field configuration data file where α = a second probe tip geometry descriptor value, a first previously stored electric field value (E_1) and a first permittivity value (ϵ_{r1}) where ϵ_{r1} is the permittivity of said thin film having approximately the same second determined thickness and known dielectric permittivity;

20 (2) reading from a second previously stored electric field configuration data file where α = a second probe tip geometry descriptor value, a second previously stored electric field value (E_2) and a second permittivity value (ϵ_{r2}) where ϵ_{r2} is the permittivity of said thin film having approximately the same second determined thickness and known dielectric permittivity;

25 (3) calculating a point on the second test sample calibration curve by solving the equation

AMENDED SHEET

PART 34 AMDT

-40-

$$\frac{\Delta f}{f} \approx \frac{\epsilon_0}{4W} \int_{V_s} (\epsilon_{r2} - \epsilon_{r1}) \vec{E}_1 \cdot \vec{E}_2 dV ; \text{and}$$

(4) repeating steps (1) through (3) until a predetermined number of points on the second test sample calibration curve have been generated.

- 5 19. An apparatus for displaying dielectric properties comprising:
- (a) a near-field scanning microwave microscope having an open-ended coaxial probe with a sharp, protruding center conductor;
 - (b) a coaxial transmission line resonator that has a resonant frequency;
 - 10 (c) a microwave source coupled to said coaxial transmission line resonator through a capacitive coupler for generating said voltage;
 - (d) a spring-loaded cantilever for supporting said sample in contact with said sharp protruding center conductor;
 - 15 (e) a bias tee coupled to said coaxial transmission line resonator for applying a local electric field to said sample in contact with said sharp protruding center conductor;
 - (f) a first motor controller for manipulating said sample in contact with said sharp protruding center conductor in a first direction;
 - 20 (g) a second motor controller for manipulating said sample in contact with said sharp protruding center conductor in a second direction;
 - (h) a third motor controller for manipulating said sample in contact with said sharp protruding center conductor in a third direction;
 - 25 (i) a coupler joined to said microwave source and said coaxial transmission line resonator;
 - (j) a detector for converting the microwave signal from said coupler into an output signal;
 - (k) a feedback circuit receives the output signal from said

AMENDED SHEET

ART 34 AMDT

-41-

detector and measures a shift change in resonance frequency, wherein said feedback circuit keeps said microwave source locked onto a predetermined resonant frequency;

(l) a processor for determining between at least one parameter related to a change in said resonant frequency and a known dielectric property value of a sample responsible for said change; wherein the processor is further able to receive from said feedback circuit said value for at least one parameter related to a change in said resonant frequency due to an unknown dielectric property of said sample and determine the value of said unknown dielectric property; and

(m) a display device for imaging the value of said unknown dielectric property once said value is determined by said processor.

20: A system for quantitatively modeling and imaging of dielectric properties, comprising:

- (a) a memory device that stores
- (i) files of electric field values, ϵ_r , and α over a predetermined range of ϵ_r and α , where ϵ_r is a dielectric permittivity value and α is representative of said probe tip geometry descriptor;
 - (ii) calibration sample resonant frequency shift values;
 - (iii) test sample resonant frequency shift values;
- (b) a model calibration curve generator that generates two model calibration curves using the stored files in said memory;
- (c) a probe calibration curve generator that generates a probe calibration curve using said generated model calibration curves and said calibration sample frequency shift values.

AMENDED SHEET

ART 34 AMDT

-42-

- (d) a test sample calibration curve generator that generates a test sample calibration curve using additional model calibration curves and additional calibration sample frequency shift values;

- 5 21. A spring-loaded sample holder comprising:
- 10 (a) a sample support having an angled end and a planar surface for supporting a sample;
- (b) a first bracing device in contact with the angled end of said sample support so as to create a pivot point;
- (c) a spring mounted to the sample support; and
- (d) a second bracing device in contact with said spring for holding the spring in place.
- 15 22. The method of claim 21, wherein a probe is held fixed and in contact with the sample.
23. The method of claim 22, wherein the sample holder and sample are scanned horizontally.
24. The method of claim 23, wherein a sensor measures the vertical displacement of the sample holder, such that this displacement is a function of the topography of the sample.
- 20 25. An apparatus for imaging a sample using a near-field scanning microwave microscope, comprising:
- a bias tee in a coaxial transmission line resonator for applying a low-frequency bias voltage to a microscope probe tip and wherein the low-frequency bias voltage is independent of a

ENCLOSURE SHEET

-43-

microwave signal, whereby a local electric field can be applied to a sample to allow for nonlinearity and tunability measuring.

- 5
26. A method for non-contact imaging of dielectric permittivity using a near-field scanning microwave microscope having a resonator with a probe tip comprising the steps of:
- (a) calibrating the near-field scanning microwave microscope using at least three dielectric samples with known dielectric properties;
 - (b) generating calibration curves;
 - 10 (c) scanning a test sample with the probe at a predetermined scanning height and generating at least one test sample frequency shift value at said predetermined scanning height; and
 - (d) determining the dielectric permittivity of the test sample based on the generated test sample resonant frequency shift value and the generated calibration curves.
- 15
27. The method of claim 26, wherein said calibrating step (a) includes the steps of:
- (i) selecting a resonant frequency of the near-field scanning microwave microscope;
 - (ii) placing one of said at least three calibration samples beneath the probe;
 - 20 (iii) positioning the probe at a first predetermined height;
 - (iv) measuring a resonant frequency at said first predetermined height;
- 25

AMENDED SHEET

12-11-2001

-44-

PART 34 AMDT

- 5
- 10
- 15
- (v) moving the probe to a second predetermined height;
 - (vi) measuring the resonant frequency at said second predetermined height;
 - (vii) calculating the difference between said resonant frequency at said first predetermined height and said resonant frequency at said second predetermined height;
 - (viii) storing in memory said calculated difference as a calibration sample resonant frequency shift value;
 - (ix) repeating steps (ii) through (viii) for each of said at least two calibration samples.

28. The method of claim 26, wherein said generating calibration curves step (b) includes the step of plotting said calibration frequency shift values as a function of dielectric constant (ϵ_r) and frequency shift.

29. The method of claim 26, wherein said scanning a test sample step (c) includes the steps of:

- 20
- 25
- (i) selecting a resonant frequency of the near-field scanning microwave microscope;
 - (ii) placing said test sample beneath the probe;
 - (iii) positioning the probe at a first predetermined height;
 - (iv) measuring a resonant frequency at said first predetermined height;
 - (v) moving the probe to a second predetermined height;

AMENDED SHEET

-45-

PART 34

- (vi) measuring the resonant frequency at said second predetermined height;
- (vii) calculating the difference between said resonant frequency at said first predetermined height and said resonant frequency at said second predetermined height;
- (viii) storing in memory said calculated difference as a test sample resonant frequency shift value.

5

5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65
66
67
68
69
70
71
72
73
74
75
76
77
78
79
80
81
82
83
84
85
86
87
88
89
90
91
92
93
94
95
96
97
98
99
100
101
102
103
104
105
106
107
108
109
110
111
112
113
114
115
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
131
132
133
134
135
136
137
138
139
140
141
142
143
144
145
146
147
148
149
150
151
152
153
154
155
156
157
158
159
160
161
162
163
164
165
166
167
168
169
170
171
172
173
174
175
176
177
178
179
180
181
182
183
184
185
186
187
188
189
190
191
192
193
194
195
196
197
198
199
200
201
202
203
204
205
206
207
208
209
210
211
212
213
214
215
216
217
218
219
220
221
222
223
224
225
226
227
228
229
230
231
232
233
234
235
236
237
238
239
240
241
242
243
244
245
246
247
248
249
250
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280
281
282
283
284
285
286
287
288
289
290
291
292
293
294
295
296
297
298
299
300
301
302
303
304
305
306
307
308
309
310
311
312
313
314
315
316
317
318
319
320
321
322
323
324
325
326
327
328
329
330
331
332
333
334
335
336
337
338
339
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
361
362
363
364
365
366
367
368
369
370
371
372
373
374
375
376
377
378
379
380
381
382
383
384
385
386
387
388
389
390
391
392
393
394
395
396
397
398
399
400
401
402
403
404
405
406
407
408
409
410
411
412
413
414
415
416
417
418
419
420
421
422
423
424
425
426
427
428
429
430
431
432
433
434
435
436
437
438
439
440
441
442
443
444
445
446
447
448
449
450
451
452
453
454
455
456
457
458
459
460
461
462
463
464
465
466
467
468
469
470
471
472
473
474
475
476
477
478
479
480
481
482
483
484
485
486
487
488
489
490
491
492
493
494
495
496
497
498
499
500
501
502
503
504
505
506
507
508
509
510
511
512
513
514
515
516
517
518
519
520
521
522
523
524
525
526
527
528
529
530
531
532
533
534
535
536
537
538
539
540
541
542
543
544
545
546
547
548
549
550
551
552
553
554
555
556
557
558
559
560
561
562
563
564
565
566
567
568
569
570
571
572
573
574
575
576
577
578
579
580
581
582
583
584
585
586
587
588
589
590
591
592
593
594
595
596
597
598
599
600
601
602
603
604
605
606
607
608
609
610
611
612
613
614
615
616
617
618
619
620
621
622
623
624
625
626
627
628
629
630
631
632
633
634
635
636
637
638
639
640
641
642
643
644
645
646
647
648
649
650
651
652
653
654
655
656
657
658
659
660
661
662
663
664
665
666
667
668
669
670
671
672
673
674
675
676
677
678
679
680
681
682
683
684
685
686
687
688
689
690
691
692
693
694
695
696
697
698
699
700
701
702
703
704
705
706
707
708
709
710
711
712
713
714
715
716
717
718
719
720
721
722
723
724
725
726
727
728
729
730
731
732
733
734
735
736
737
738
739
740
741
742
743
744
745
746
747
748
749
750
751
752
753
754
755
756
757
758
759
760
761
762
763
764
765
766
767
768
769
770
771
772
773
774
775
776
777
778
779
780
781
782
783
784
785
786
787
788
789
790
791
792
793
794
795
796
797
798
799
800
801
802
803
804
805
806
807
808
809
810
811
812
813
814
815
816
817
818
819
820
821
822
823
824
825
826
827
828
829
830
831
832
833
834
835
836
837
838
839
840
841
842
843
844
845
846
847
848
849
850
851
852
853
854
855
856
857
858
859
860
861
862
863
864
865
866
867
868
869
870
871
872
873
874
875
876
877
878
879
880
881
882
883
884
885
886
887
888
889
890
891
892
893
894
895
896
897
898
899
900
901
902
903
904
905
906
907
908
909
910
911
912
913
914
915
916
917
918
919
920
921
922
923
924
925
926
927
928
929
930
931
932
933
934
935
936
937
938
939
940
941
942
943
944
945
946
947
948
949
950
951
952
953
954
955
956
957
958
959
960
961
962
963
964
965
966
967
968
969
970
971
972
973
974
975
976
977
978
979
980
981
982
983
984
985
986
987
988
989
990
991
992
993
994
995
996
997
998
999
1000
1001
1002
1003
1004
1005
1006
1007
1008
1009
1010
1011
1012
1013
1014
1015
1016
1017
1018
1019
1020
1021
1022
1023
1024
1025
1026
1027
1028
1029
1030
1031
1032
1033
1034
1035
1036
1037
1038
1039
1040
1041
1042
1043
1044
1045
1046
1047
1048
1049
1050
1051
1052
1053
1054
1055
1056
1057
1058
1059
1060
1061
1062
1063
1064
1065
1066
1067
1068
1069
1070
1071
1072
1073
1074
1075
1076
1077
1078
1079
1080
1081
1082
1083
1084
1085
1086
1087
1088
1089
1090
1091
1092
1093
1094
1095
1096
1097
1098
1099
1100
1101
1102
1103
1104
1105
1106
1107
1108
1109
1110
1111
1112
1113
1114
1115
1116
1117
1118
1119
1120
1121
1122
1123
1124
1125
1126
1127
1128
1129
1130
1131
1132
1133
1134
1135
1136
1137
1138
1139
1140
1141
1142
1143
1144
1145
1146
1147
1148
1149
1150
1151
1152
1153
1154
1155
1156
1157
1158
1159
1160
1161
1162
1163
1164
1165
1166
1167
1168
1169
1170
1171
1172
1173
1174
1175
1176
1177
1178
1179
1180
1181
1182
1183
1184
1185
1186
1187
1188
1189
1190
1191
1192
1193
1194
1195
1196
1197
1198
1199
1200
1201
1202
1203
1204
1205
1206
1207
1208
1209
1210
1211
1212
1213
1214
1215
1216
1217
1218
1219
1220
1221
1222
1223
1224
1225
1226
1227
1228
1229
1230
1231
1232
1233
1234
1235
1236
1237
1238
1239
1240
1241
1242
1243
1244
1245
1246
1247
1248
1249
1250
1251
1252
1253
1254
1255
1256
1257
1258
1259
1260
1261
1262
1263
1264
1265
1266
1267
1268
1269
1270
1271
1272
1273
1274
1275
1276
1277
1278
1279
1280
1281
1282
1283
1284
1285
1286
1287
1288
1289
1290
1291
1292
1293
1294
1295
1296
1297
1298
1299
1300
1301
1302
1303
1304
1305
1306
1307
1308
1309
1310
1311
1312
1313
1314
1315
1316
1317
1318
1319
1320
1321
1322
1323
1324
1325
1326
1327
1328
1329
1330
1331
1332
1333
1334
1335
1336
1337
1338
1339
1340
1341
1342
1343
1344
1345
1346
1347
1348
1349
1350
1351
1352
1353
1354
1355
1356
1357
1358
1359
1360
1361
1362
1363
1364
1365
1366
1367
1368
1369
1370
1371
1372
1373
1374
1375
1376
1377
1378
1379
1380
1381
1382
1383
1384
1385
1386
1387
1388
1389
1390
1391
1392
1393
1394
1395
1396
1397
1398
1399
1400
1401
1402
1403
1404
1405
1406
1407
1408
1409
1410
1411
1412
1413
1414
1415
1416
1417
1418
1419
1420
1421
1422
1423
1424
1425
1426
1427
1428
1429
1430
1431
1432
1433
1434
1435
1436
1437
1438
1439
1440
1441
1442
1443
1444
1445
1446
1447
1448
1449
1450
1451
1452
1453
1454
1455
1456
1457
1458
1459
1460
1461
1462
1463
1464
1465
1466
1467
1468
1469
1470
1471
1472
1473
1474
1475
1476
1477
1478
1479
1480
1481
1482
1483
1484
1485
1486
1487
1488
1489
1490
1491
1492
1493
1494
1495
1496
1497
1498
1499
1500
1501
1502
1503
1504
1505
1506
1507
1508
1509
1510
1511
1512
1513
1514
1515
1516
1517
1518
1519
1520
1521
1522
1523
1524
1525
1526
1527
1528
1529
1530
1531
1532
1533
1534
1535
1536
1537
1538
1539
1540
1541
1542
1543
1544
1545
1546
1547
1548
1549
1550
1551
1552
1553
1554
1555
1556
1557
1558
1559
1560
1561
1562
1563
1564
1565
1566
1567
1568
1569
1570
1571
1572
1573
1574
1575
1576
1577
1578
1579
1580
1581
1582
1583
1584
1585
1586
1587
1588
1589
1590
1591
1592
1593
1594
1595
1596
1597
1598
1599
1600
1601
1602
1603
1604
1605
1606
1607
1608
1609
1610
1611
1612
1613
1614
1615
1616
1617
1618
1619
1620
1621
1622
1623
1624
1625
1626
1627
1628
1629
1630
1631
1632
1633
1634
1635
1636
1637
1638
1639
1640
1641
1642
1643
1644
1645
1646
1647
1648
1649
1650
1651
1652
1653
1654
1655
1656
1657
1658
1659
1660
1661
1662
1663
1664
1665
1666
1667
1668
1669
1670
1671
1672
1673
1674
1675
1676
1677
1678
1679
1680
1681
1682
1683
1684
1685
1686
1687
1688
1689
1690
1691
1692
1693
1694
1695
1696
1697
1698
1699
1700
1701
1702
1703
1704
1705
1706
1707
1708
1709
1710
1711
1712
1713
1714
1715
1716
1717
1718
1719
1720
1721
1722
1723
1724
1725
1726
1727
1728
1729
1730
1731
1732
1733
1734
1735
1736
1737
1738
1739
1740
1741
1742
1743
1744
1745
1746
1747
1748
1749
1750
1751
1752
1753
1754
1755
1756
1757
1758
1759
1760
1761
1762
1763
1764
1765
1766
1767
1768
1769
1770
1771
1772
1773
1774
1775
1776
1777
1778
1779
1780
1781
1782
1783
1784
1785
1786
1787
1788
1789
1790
1791
1792
1793
1794
1795
1796
1797
1798
1799
1800
1801
1802
1803
1804
1805
1806
1807
1808
1809
1810
1811
1812
1813
1814
1815
1816
1817
1818
1819
1820
1821
1822
1823
1824
1825
1826
1827
1828
1829
1830
1831
1832
1833
1834
1835
1836
1837
1838
1839
1840
1841
1842
1843
1844
1845
1846
1847
1848
1849
1850
1851
1852
1853
1854
1855
1856
1857
1858
1859
1860
1861
1862
1863
1864
1865
1866
1867
1868
1869
1870
1871
1872
1873
1874
1875
1876
1877
1878
1879
1880
1881
1882
1883
1884
1885
1886
1887
1888
1889
1890
1891
1892
1893
1894
1895
1896
1897
1898
1899
1900
1901
1902
1903
1904
1905
1906
1907
1908
1909
1910
1911
1912
1913
1914
1915
1916
1917
1918
1919
1920
1921
1922
1923
1924
1925
1926
1927
1928
1929
1930
1931
1932
1933
1934
1935
1936
1937
1938
1939
1940
1941
1942
1943
1944
1945
1946
1947
1948
1949
1950
1951
1952
1953
1954
1955
1956
1957
1958
1959
1960
1961
1962
1963
1964
1965
1966
1967
1968
1969
1970
1971
1972
1973
1974
1975
1976
1977
1978
1979
1980
1981
1982
1983
1984
1985
1986
1987
1988
1989
1990
1991
1992
1993
1994
1995
1996
1997
1998
1999
2000
2001
2002
2003
2004
2005
2006
2007
2008
2009
2010
2011
2012
2013
2014
2015
2016
2017
2018
2019
2020
2021
2022
2023
2024
2025
2026
2027
2028
2029
2030
2031
2032
2033
2034
2035
2036
2037
2038
2039
2040
2041
2042
2043
2044
2045
2046
2047
2048
2049
2050
2051
2052
2053
2054
2055
2056
2057
2058
2059
2060
2061
2062
2063
2064
2065
2066
2067
2068
2069
2070
2071
2072
2073
2074
2075
2076
2077
2078
2079
2080
2081
2082
2083
2084
2085
2086
2087
2088
2089
2090
2091
2092
2093
2094
2095
2096
2097
2098
2099
2100
2101
2102
2103
2104
2105
2106
2107
2108
2109
2110
2111
2112
2113
2114
2115
2116
2117
2118
2119
2120
2121
2122
2123
2124
2125
2126
2127
2128
2129
2130
2131
2132
2133
2134
2135
2136
2137
2138
2139
2140
2141
2142
2143
2144
2145
2146
2147
2148
2149
2150
2151
2152
2153
2154
2155
2156
2157
2158
2159
2160
2161
2162
2163
2164
2165
2166
2167
2168
2169
2170
2171
2172
2173
2174
2175
2176
2177
2178
2179
2180
2181
2182
2183
2184
2185
2186
2187
2188
2189
2190
2191
2192
2193
2194

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
22 March 2001 (22.03.2001)

PCT

(10) International Publication Number
WO 01/20352 A1

(51) International Patent Classification⁷: **G01R 27/26**

(74) Agents: SOKOHL, Robert, E. et al.; Sterne, Kessler, Goldstein & Fox P.L.L.C., Suite 600, 1100 New York Avenue, N.W., Washington, DC 20005-3934 (US).

(21) International Application Number: PCT/US00/08943

(22) International Filing Date: 5 April 2000 (05.04.2000)

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

60/153,354 10 September 1999 (10.09.1999) US
60/191,903 24 March 2000 (24.03.2000) US

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

(71) Applicants and

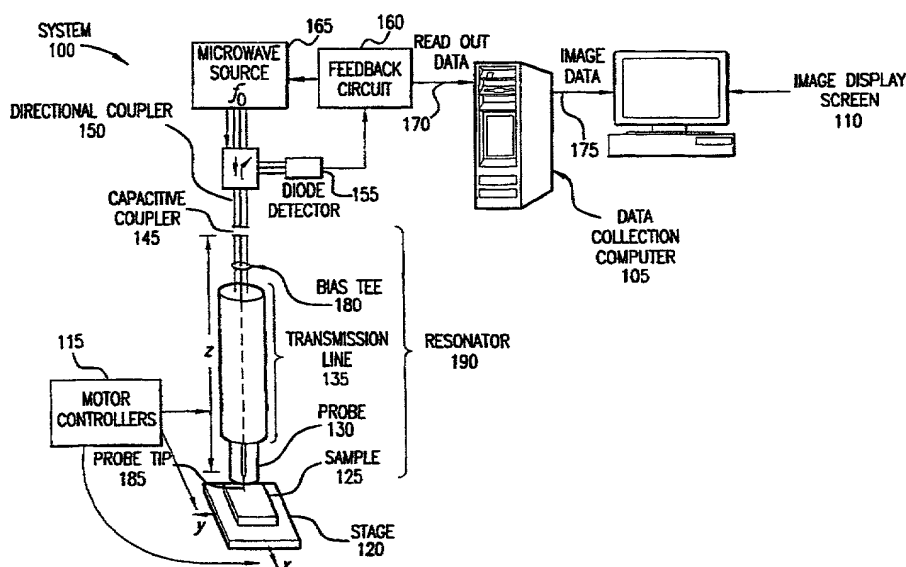
(72) Inventors: ANLAGE, Steven, Mark [US/US]; 8810 Herons Flight, Laurel, MD 20723 (US). STEINHAUER, David, Ethan [US/US]; 1121 Westview Terrace, Laurel, MD 20707 (US). VLAHACOS, Constantine, P. [US/US]; 3014 East Baltimore Street, Baltimore, MD 21224 (US). WELLSTOOD, Frederick, C. [US/US]; 6123 Main Street, Lanham, MD 20706 (US).

Published:

- With international search report.
- With a statement concerning non-prejudicial disclosure or exception to lack of novelty.

[Continued on next page]

(54) Title: QUANTITATIVE IMAGING OF DIELECTIC PERMITTIVITY AND TUNABILITY



(57) Abstract: A near-field scanning microwave microscope images the permittivity and dielectric tunability of bulk and thin film dielectric samples on a length scale of about 1 micron or less. The microscope is sensitive to the linear permittivity, as well as to nonlinear dielectric terms, which can be measured as a function of an applied electric field. A versatile finite element model is used for the system, which allows quantitative results to be obtained. The technique is non destructive and has broadband (0.1-50 GHz) capability.

1/19

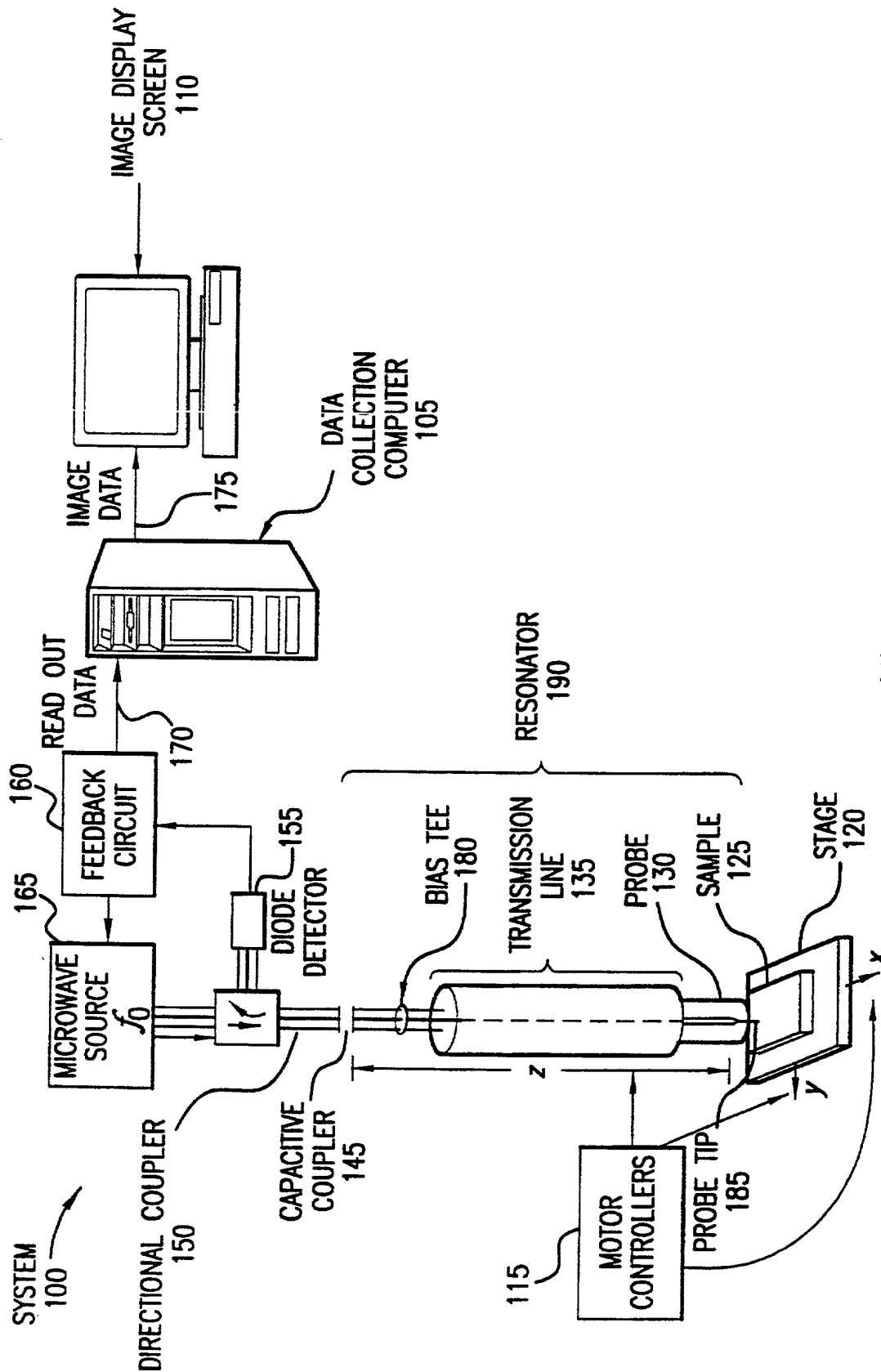


FIG.1

2/19

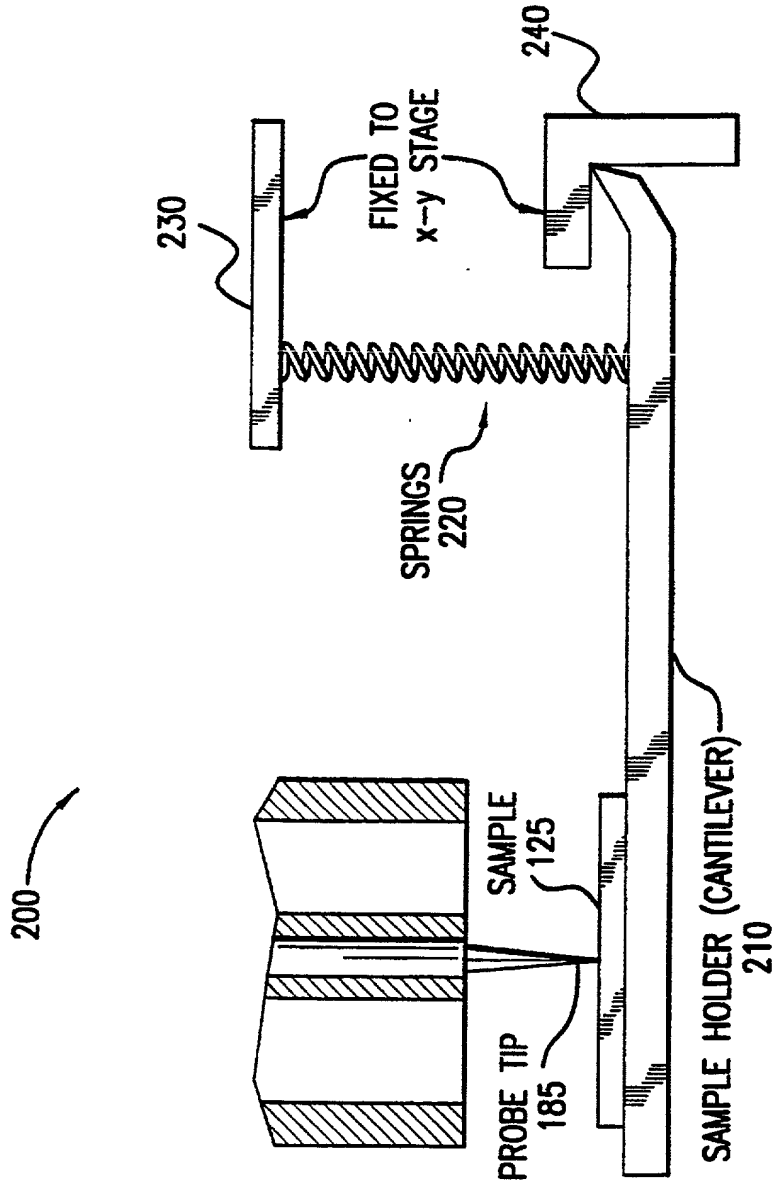


FIG.2

3/19

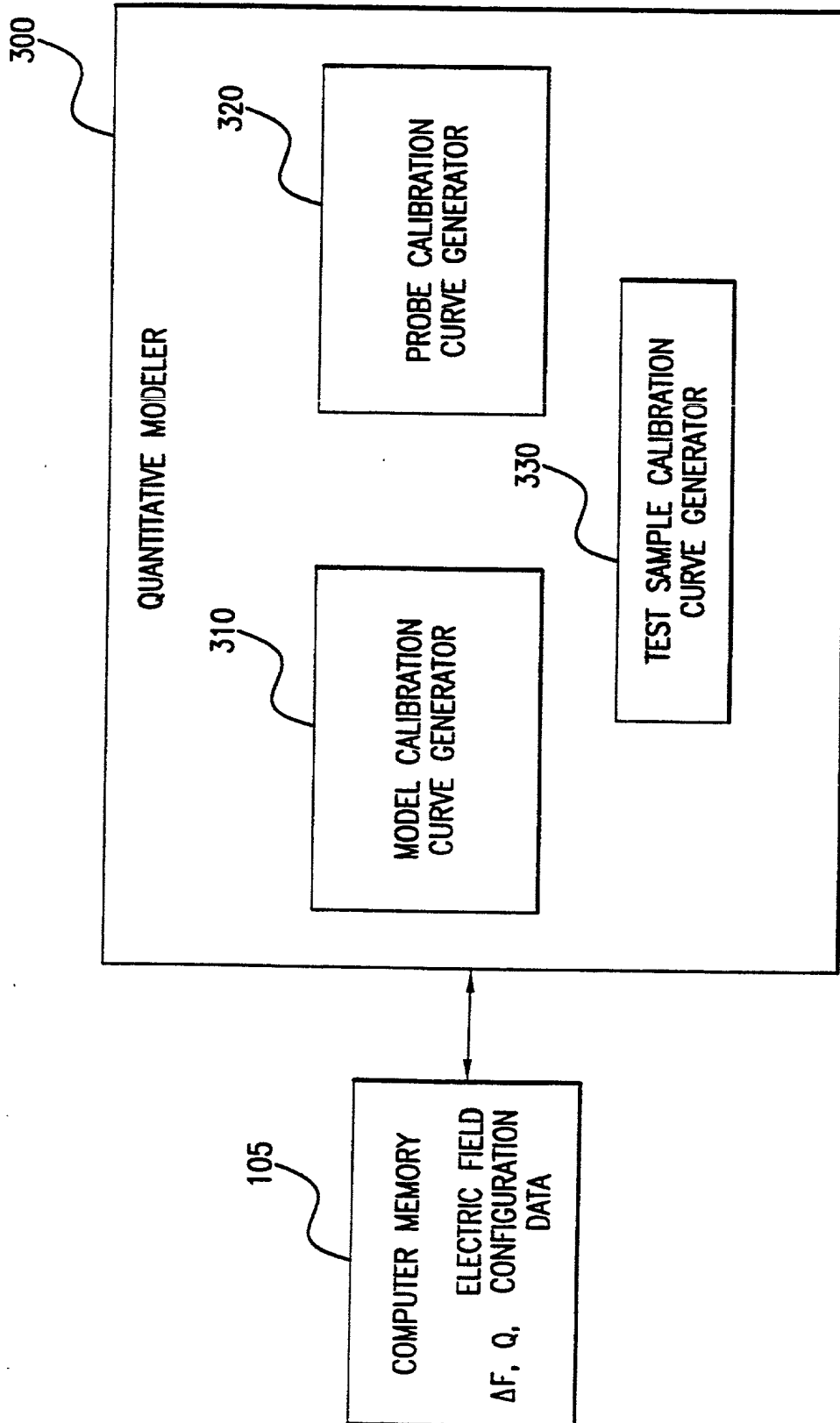


FIG.3

4/19

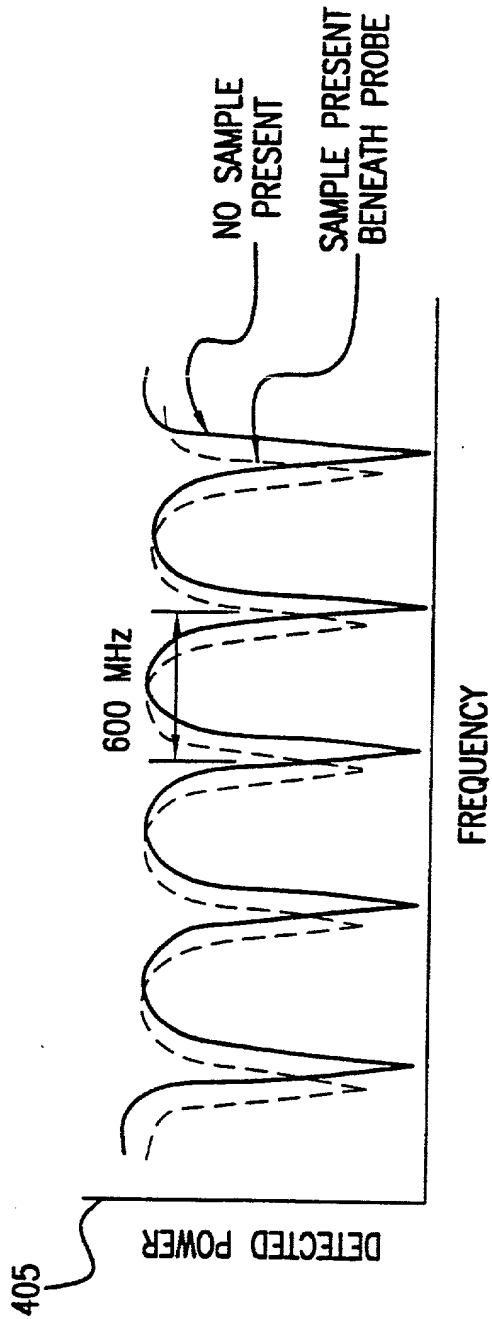


FIG. 4

PERTURBATION FORMULA

$$\frac{\Delta f}{f} \approx \frac{(\epsilon_{r1} - \epsilon_{r2})}{4W} \int_{V_S} \vec{E}_1 \cdot \vec{E}_2 \, dV$$

$$\Delta f = f(\epsilon_{r1}, \epsilon_{r2})$$

FIG. 5

5/19

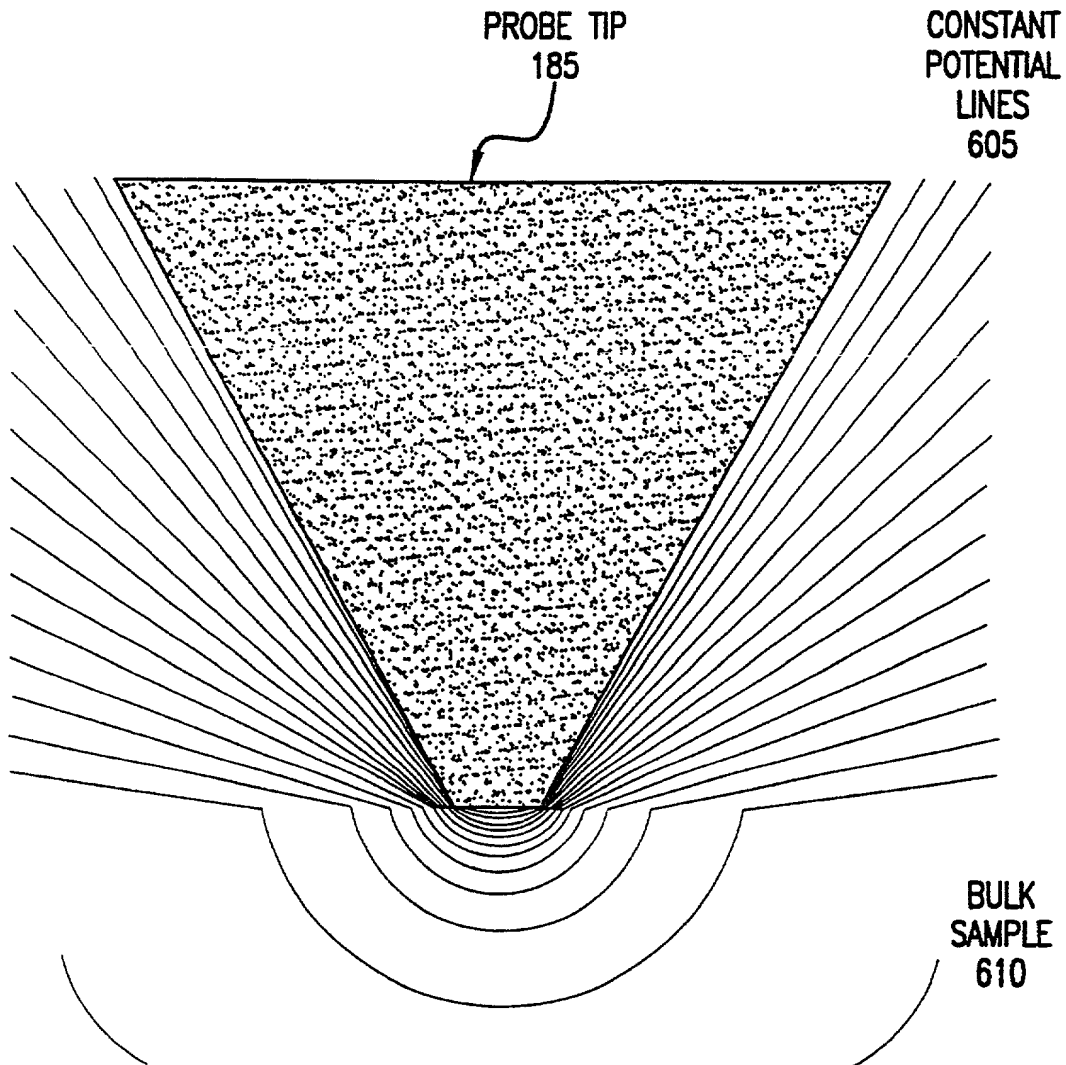


FIG.6

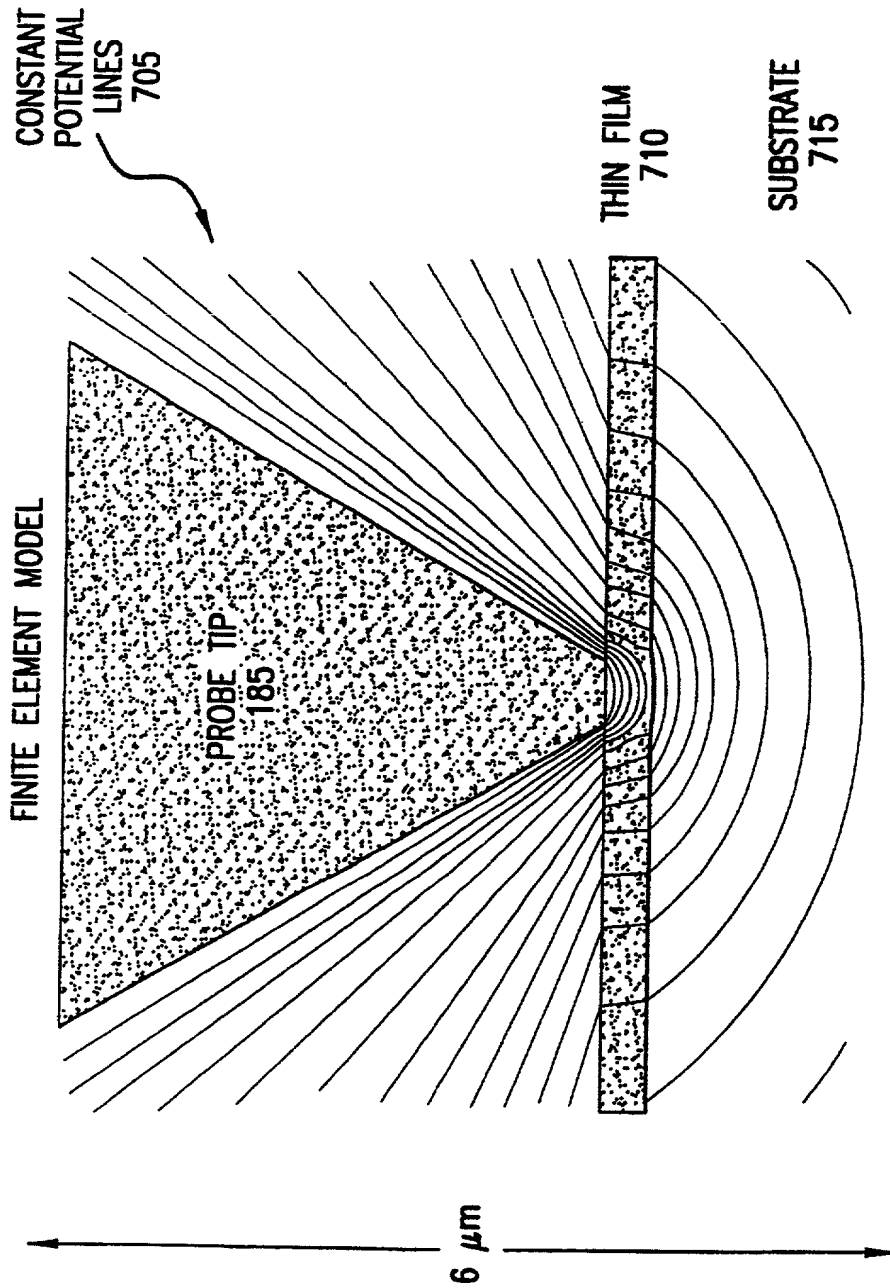
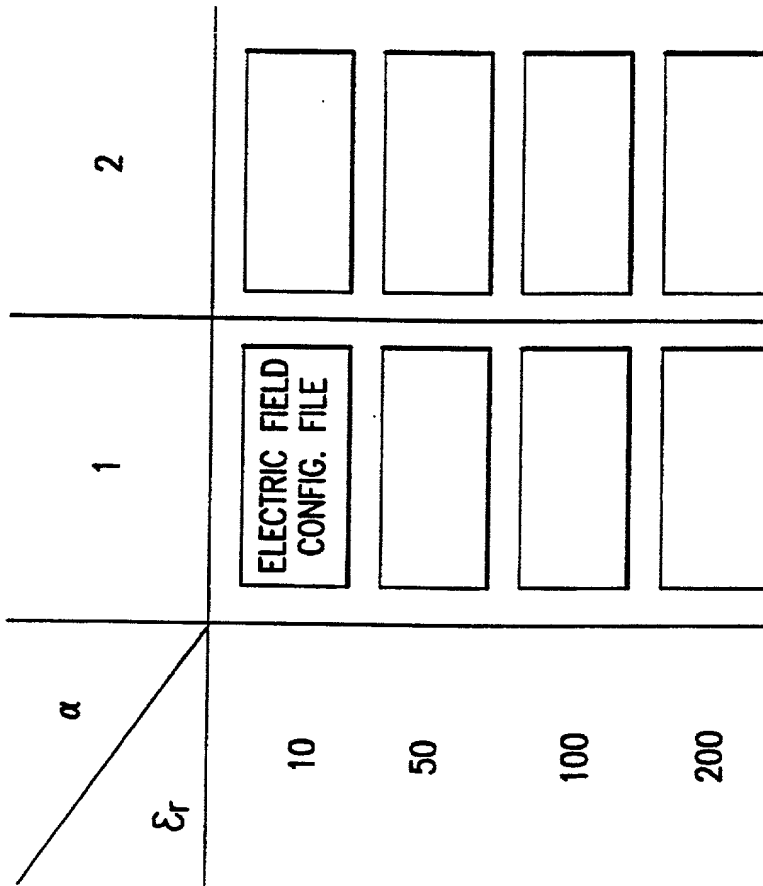


FIG.7

7/19

$t=0.5$



ELECTRIC FIELD
CONFIGURATION DATA
FILE CONTENTS

ϵ_r	t	α
$\phi(r,z)$		

FIG.8

10/669996

8/19

ROUTINE FOR DETERMINING DIELECTRIC PROPERTIES

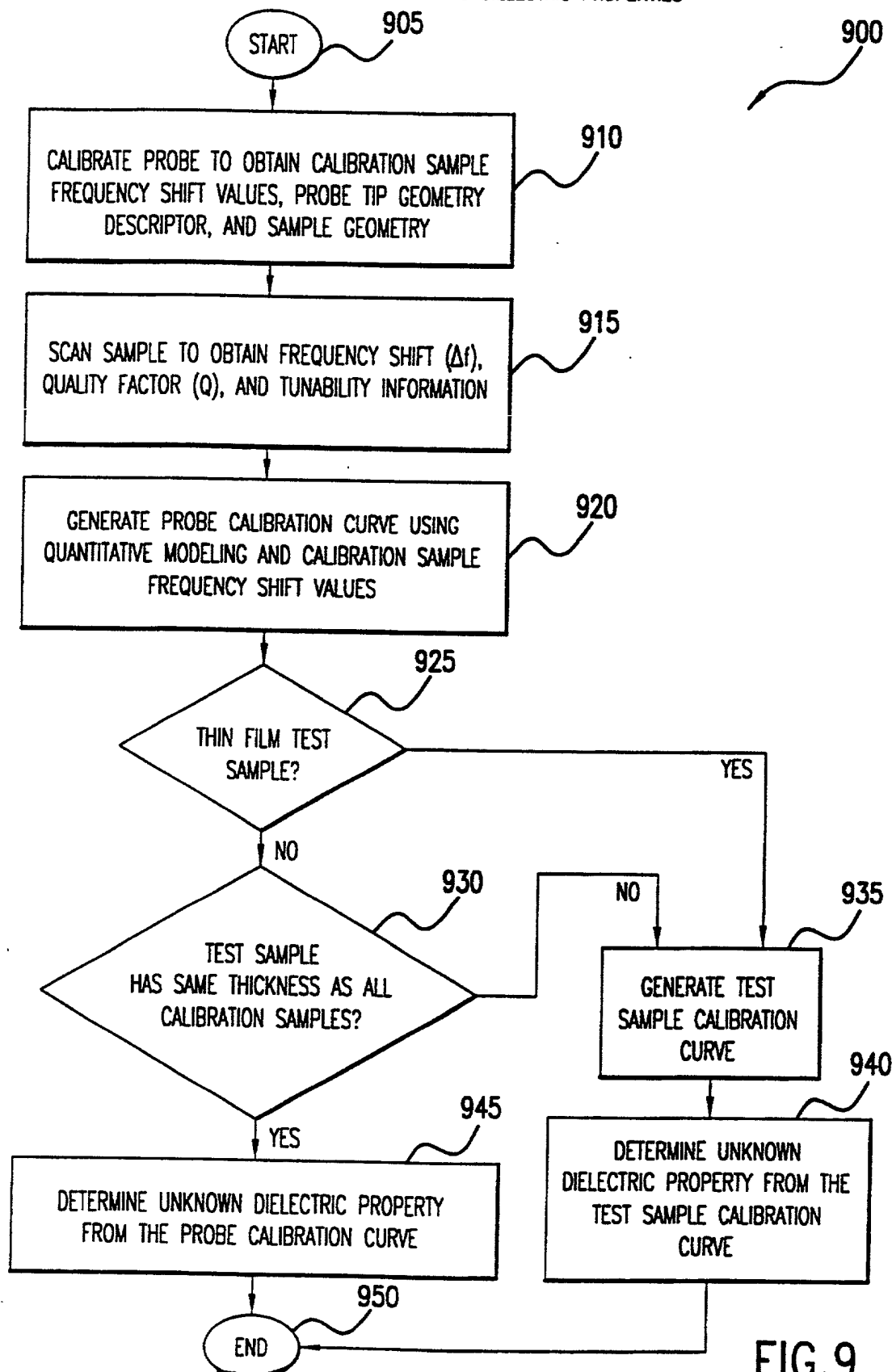


FIG.9

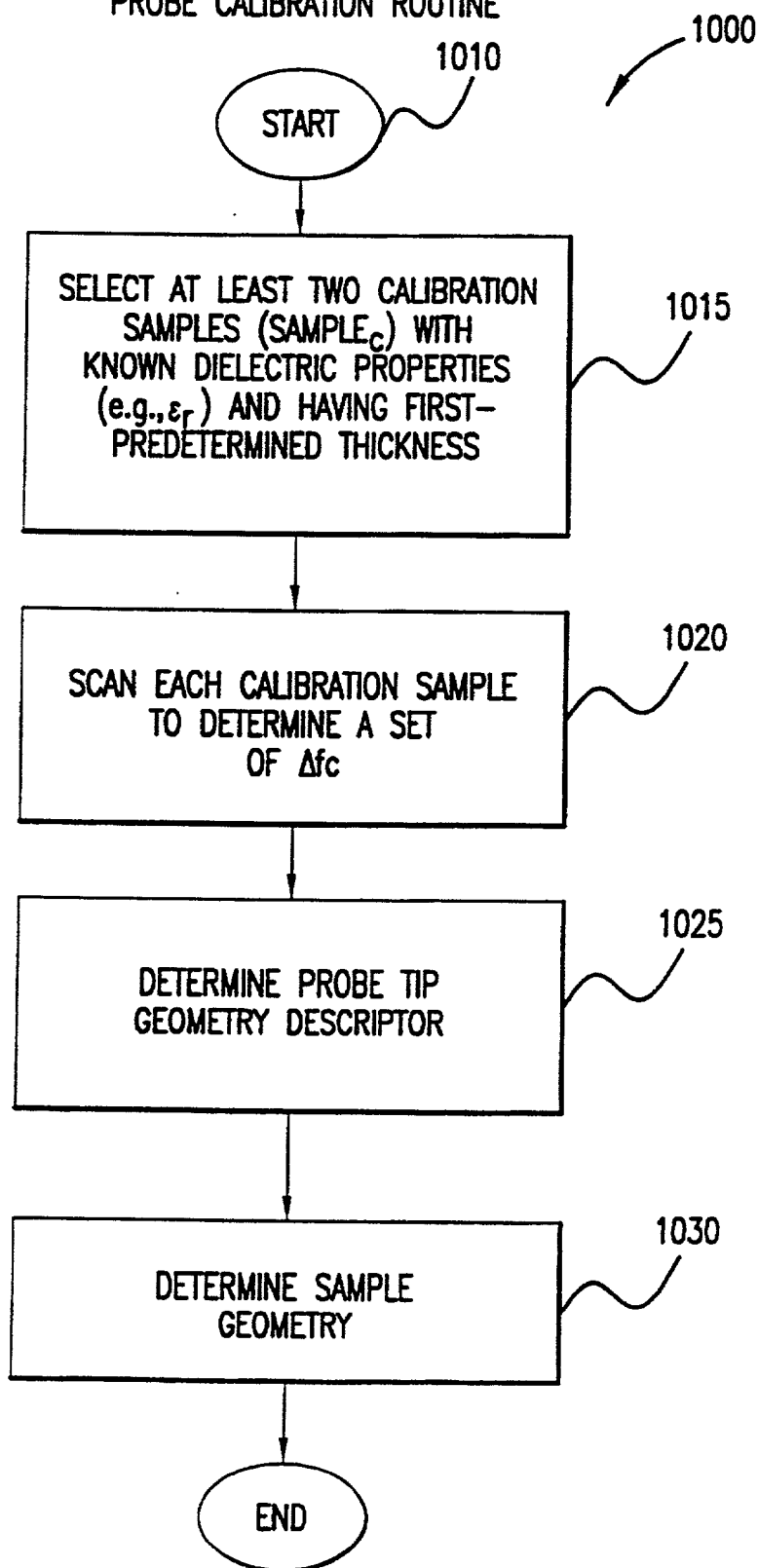
9/19
PROBE CALIBRATION ROUTINE

FIG. 10

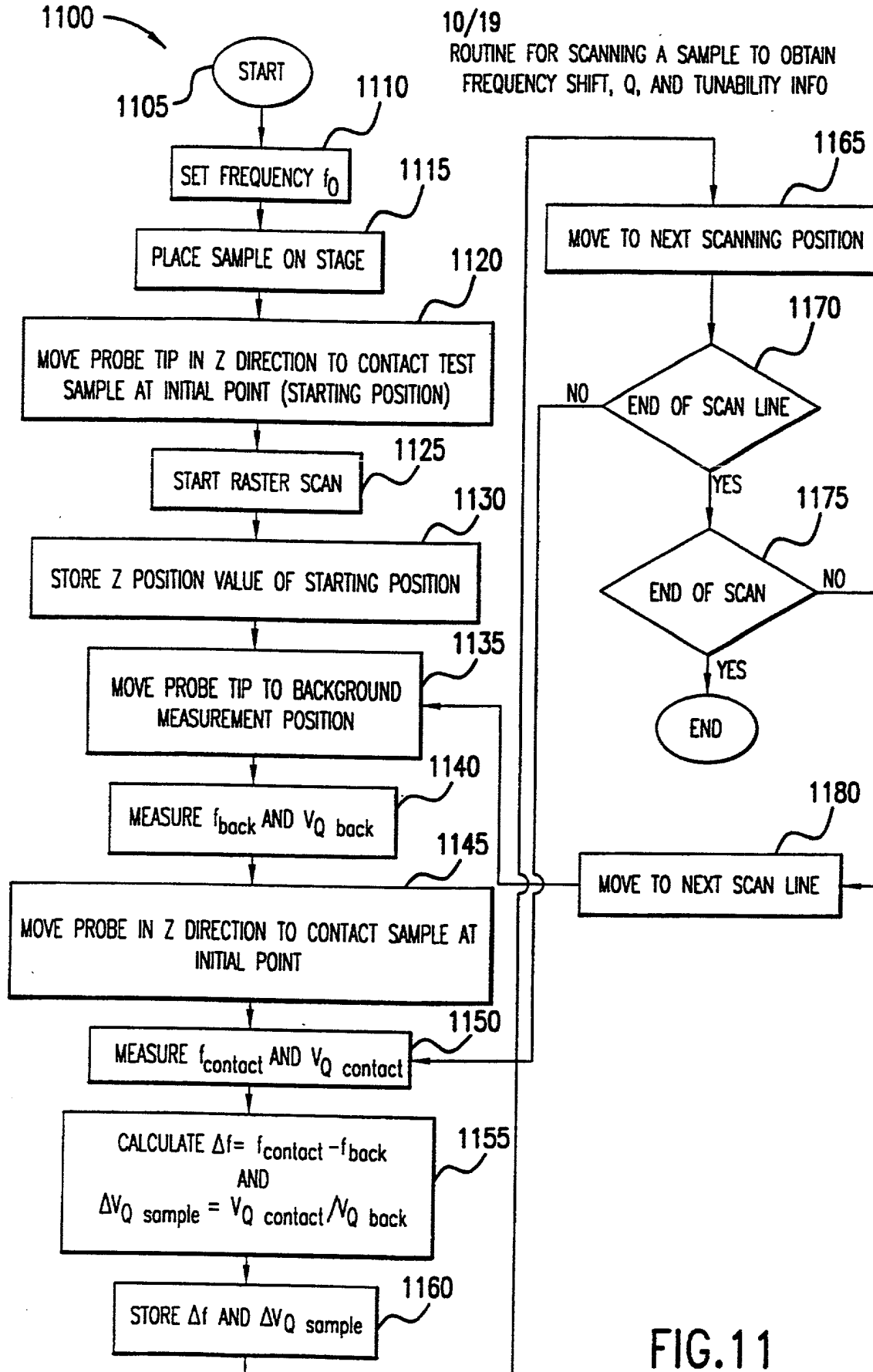


FIG.11

11/19

ROUTINE FOR GENERATING A CALIBRATION CURVE

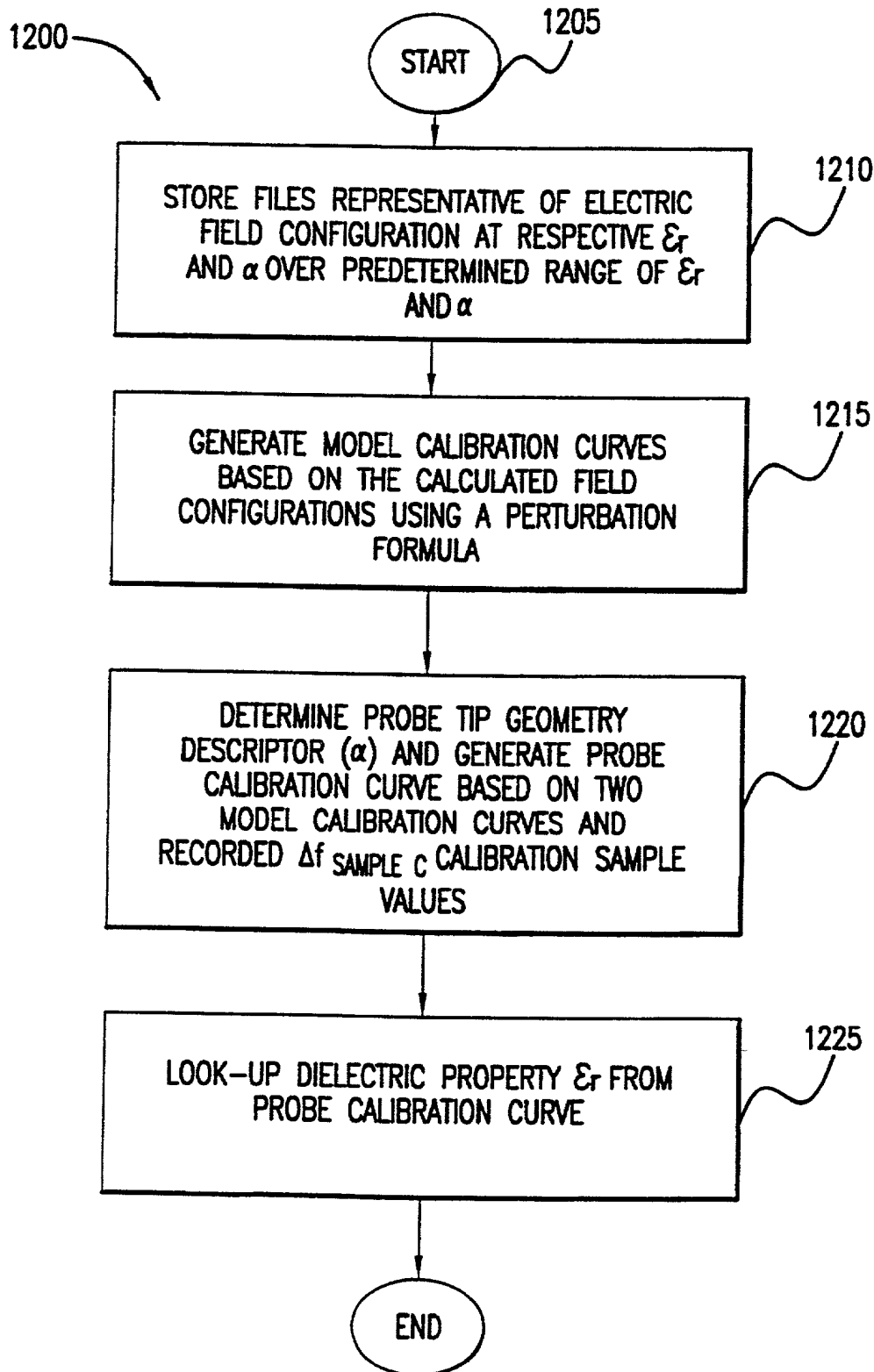


FIG.12

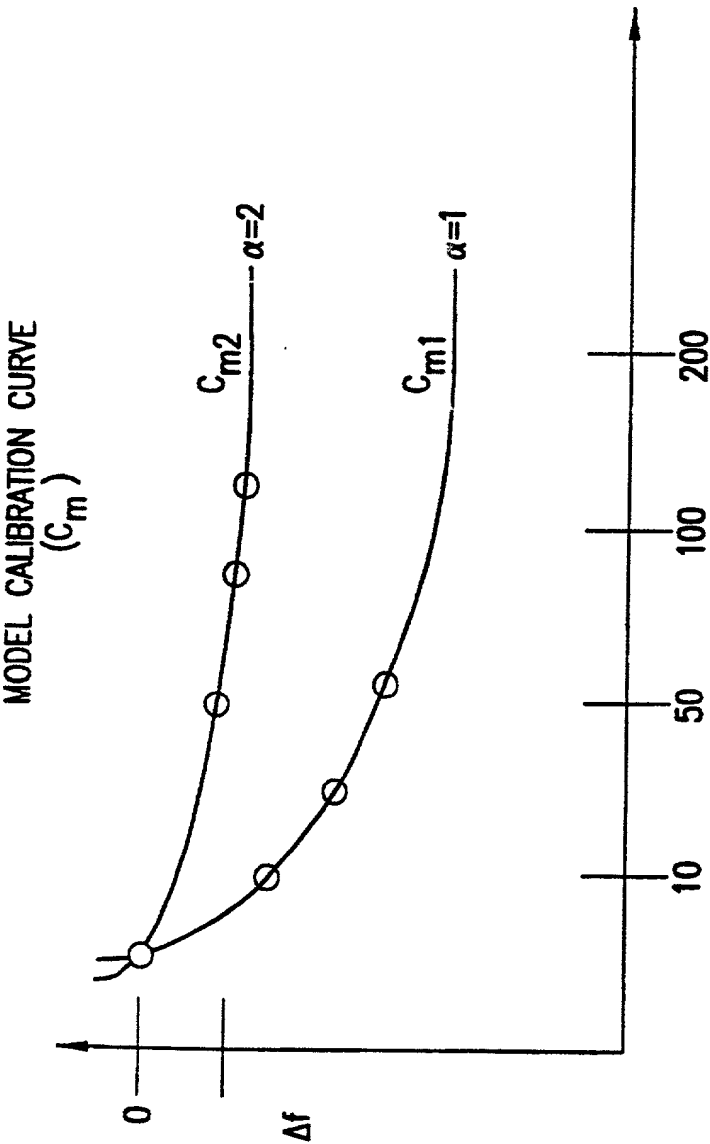


FIG.13

13/19

PROBE CALIBRATION CURVE
(C_p)

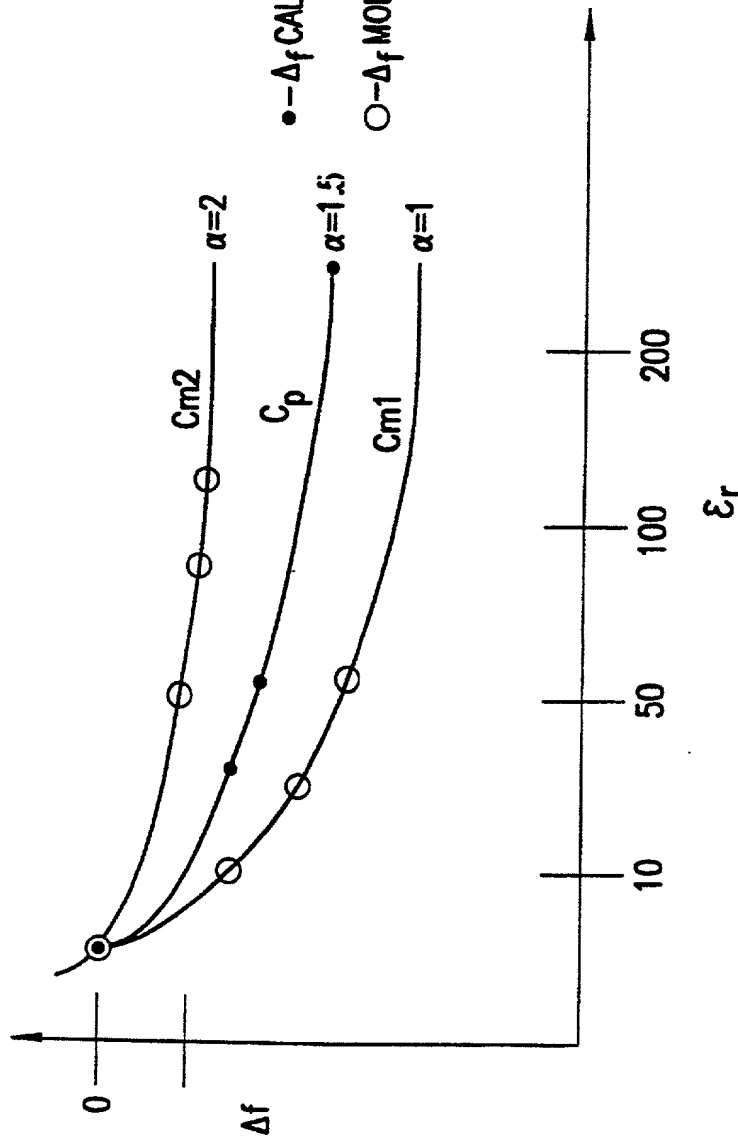


FIG.14

14/19

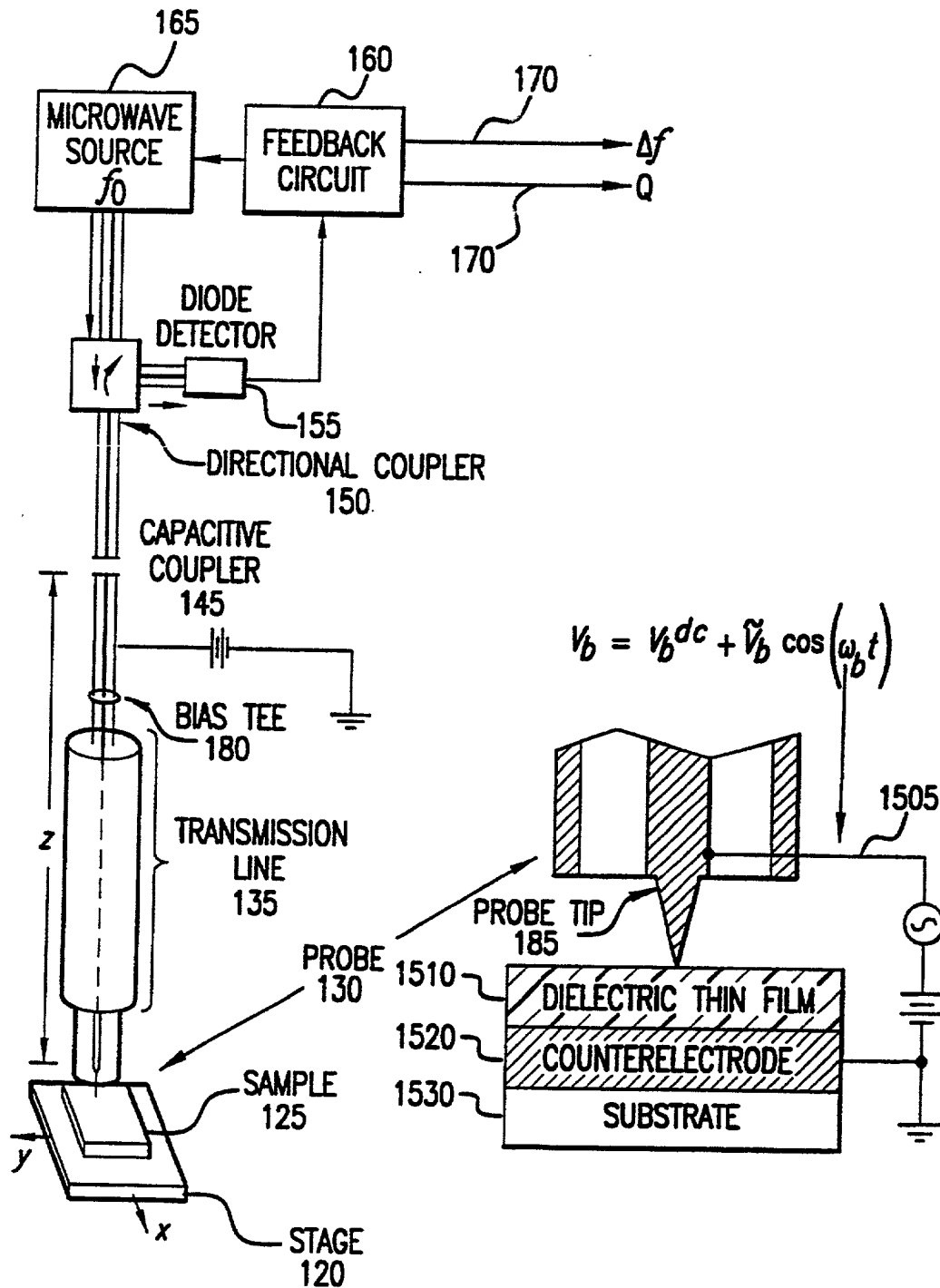
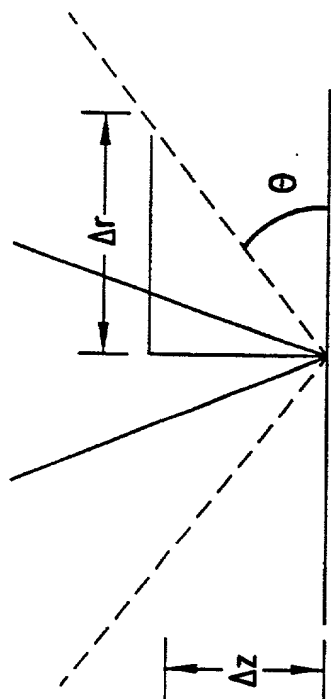


FIG.15



SMALL ASPECT RATIO = BLUNT TIP

$$\text{ASPECT RATIO} \equiv \alpha = \frac{\Delta z}{\Delta r} = \tan \theta$$

HIGH ASPECT RATIO = SHARP TIP

FIG.16

TEST SAMPLE CALIBRATION CURVE

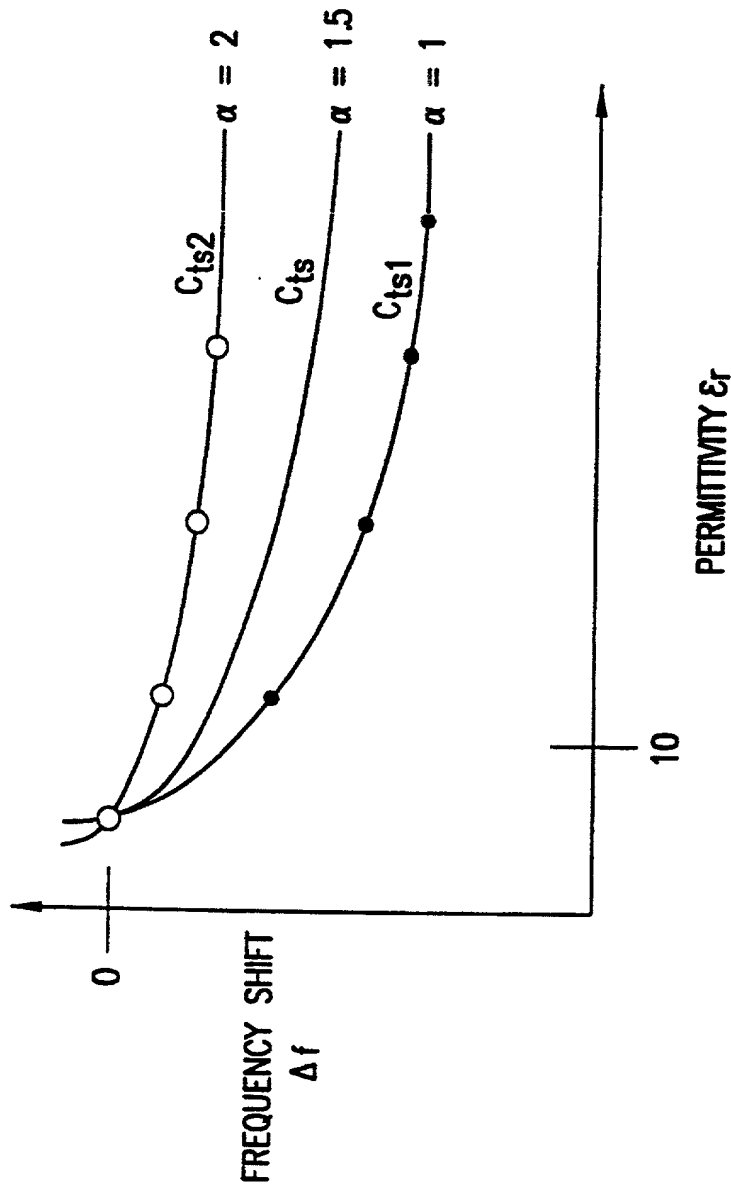


FIG.17

17/19

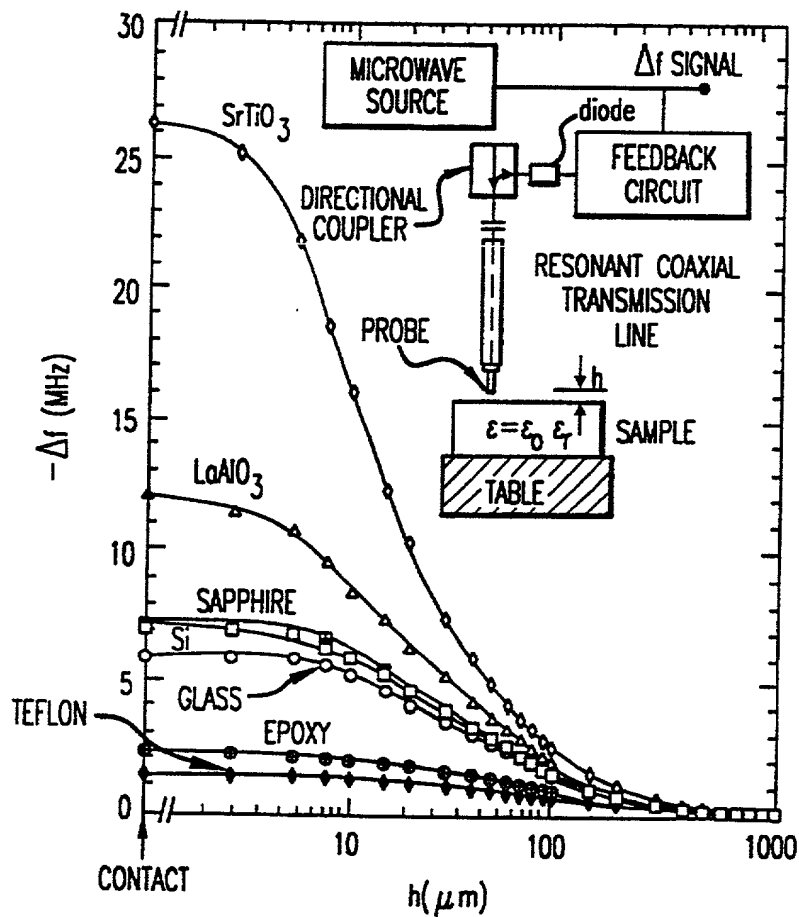


FIG. 18

18/19

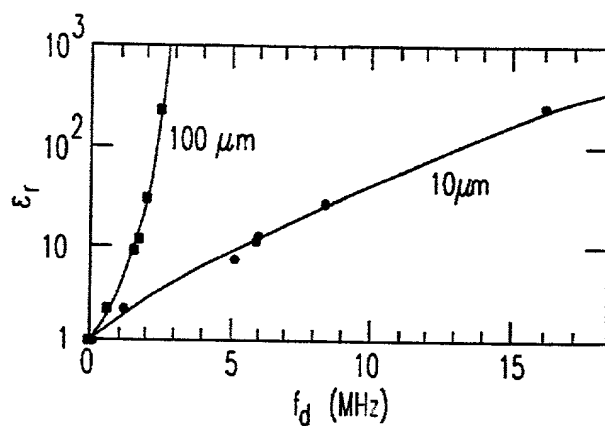


FIG. 19

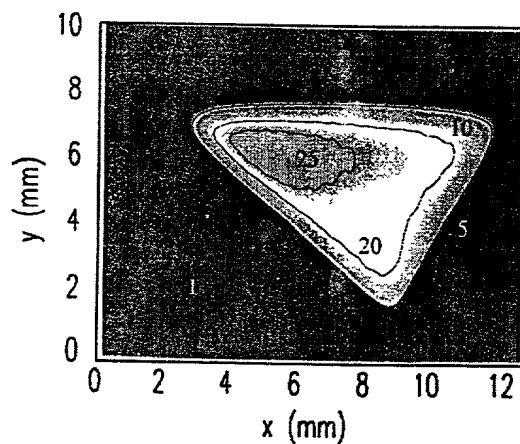


FIG. 20

MATERIAL	EXPERIMENTAL VALUE	LITERATURE VALUE	FREQUENCY (GHz)	REFERENCES
SILICON	12	11.7	100	11
GLASS	12	6.7	10^{-3}	12
SrTiO ₃	180	230	0.1	9
LaAlO ₃	20	23.9	18	10
SAPPHIRE				
(CERAMIC)	20	10.0	100	11
TEFLON	2.1	2.1	10	9

FIG. 21

19/19

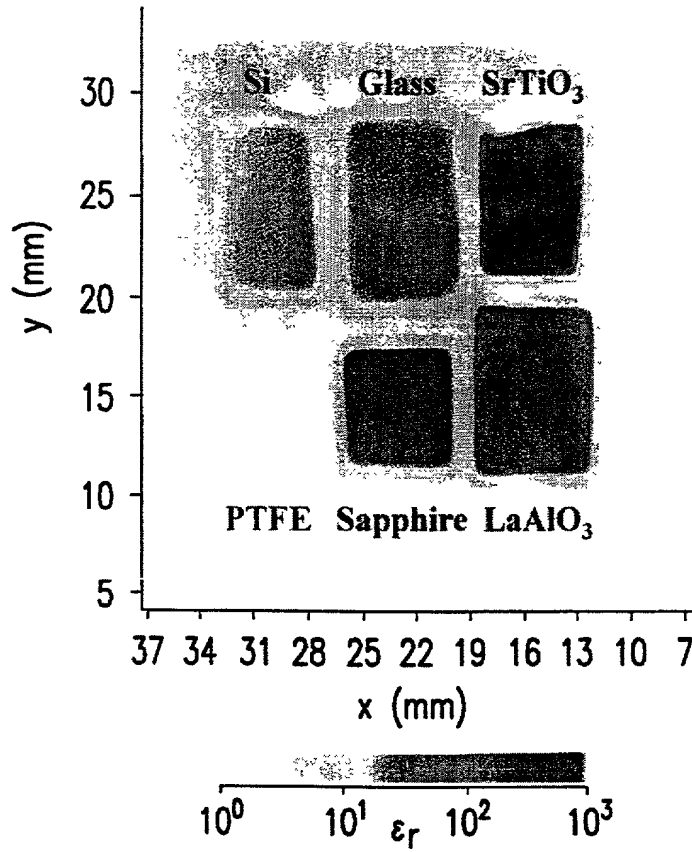


FIG. 22A

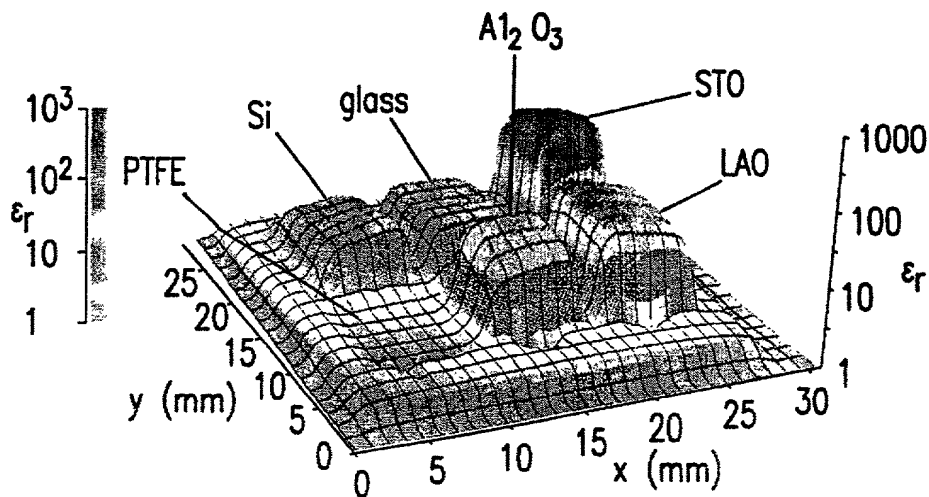


FIG. 22B

DECLARATION FOR PATENT APPLICATION

Docket Number (Optional)

MR2833-14

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled QUANTITATIVE IMAGING OF DIELECTRIC PERMITTIVITY AND TUNABILITY, the specification of which is attached hereto unless the following box is checked:

☐ was filed on _____ as United States Application Number or PCT International Application Number _____ and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

PCT/US00/08943 ✓ (Number)	PCT ✓ (Country)	5 April 2000 ✓ (Day/Month/Year Filed)	<input type="checkbox"/>
_____ (Number)	_____ (Country)	_____ (Day/Month/Year Filed)	<input type="checkbox"/>

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

60/153,354 ✓ (Application Number)	10 September 1999 ✓ (Filing Date)
60/191,903 ✓ (Application Number)	24 March 2000 ✓ (Filing Date)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

(Application Number)	(Filing Date)	(Status-patented, pending, abandoned)
----------------------	---------------	---------------------------------------

(Application Number)	(Filing Date)	(Status-patented, pending, abandoned)
----------------------	---------------	---------------------------------------

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

MORTON J. ROSENBERG, ESQ., REG. # 26,049;
DAVID I. KLEIN, ESQ., REG. # 33,253; JUN Y. LEE, ESQ., REG. # 40,262
MORTON J. ROSENBERG at telephone number 410-465-6678
ROSENBERG, KLEIN & LEE
3458 ELLICOTT CENTER DRIVE SUITE 101
ELLICOTT CITY, MD 21043
FAX #: 410-461-3067
E-MAIL ADDRESS: rkl@rklpatlaw.com

Address all telephone calls to
Address all correspondence to

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor (given name, family name) Steven Mark Anlage (Anlage is Family Name)

Inventor's signature _____ Date 2/28/02

Citizenship United States

Mailing Address 8810 Herons Flight
Laurel, Maryland 20723 MD.

Full name of second joint inventor, if any (given name, family name) David E. Steinhauer (Steinhauer is Family Name)

Second Inventor's signature _____ Date 26-Feb-2002

Citizenship United States

Mailing Address 2941 - 217th Place SW
Brier, Washington 98036 D.C.

☒ Additional Inventors are being named on separately numbered sheets attached hereto.

3-00

Third Joint Inventor	<u>Constantine P. Vlahacos</u> (Vlahacos is Family Name)	
Inventor's Signature	<u>Constantine P. Vlahacos</u>	Date 28-Feb. 2002
Citizenship	United States of America —	
Mailing Address	3014 E. Baltimore Street	
	<u>Baltimore</u> , Maryland 21224 MD.	

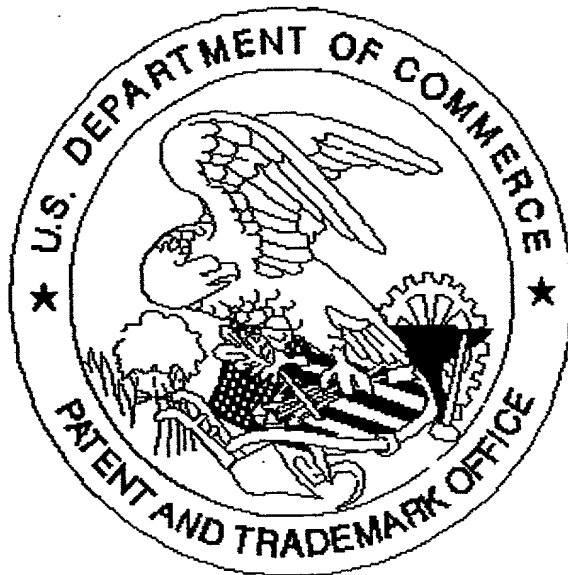
4-00

Fourth Joint Inventor	<u>Frederick C. Wellstood</u> (Wellstood is Family Name)	
Inventor's Signature	<u>F. C. Wellstood</u>	Date 2/28/2002
Citizenship	United States —	
Mailing Address	6123 Main Street	
	<u>Lanham</u> , Maryland 20706 MD.	

Fifth Joint Inventor		
Inventor's Signature		Date
Citizenship		
Mailing Address		

Sixth Joint Inventor		
Inventor's Signature		Date
Citizenship		
Mailing Address		

United States Patent & Trademark Office
Office of Initial Patent Examination -- Scanning Division



Application deficiencies found during scanning:

☐ Page(s) _____ of _____ were not present
for scanning. (Document title)

☐ Page(s) _____ of _____ were not present
for scanning. (Document title)

✓ Scanned copy is best available. Some drawings are
too dark.